

FINAL REPORT

PROGRAM FOR SEA LEVEL TEST FIRING OF ROCKET ENGINES

(Title Unclassified)

FEBRUARY 1965

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REPORT NO. S-454

THE MARQUARDT CORPORATION



PROGRAM FOR SEA LEVEL TEST FIRING OF ROCKET ENGINES (Title Unclassified)

FOR THE PERIOD

1 SEPTEMBER 1964 THROUGH 1 FEBRUARY 1965

CONTRACT NO.: NAS 9-3465
TMC PROJECT 641

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I. INTRODUCTION

This report presents the results in the testing of advanced material concepts as applied to rocket engine chambers under NASA Contract 9-3465 during the period from 1 September 1964 through 1 February 1965.

II. <u>SUMMARY</u>

A contract was received at The Marquardt Corporation on 1 September 1965 to evaluate an advanced high temperature coating for radiation cooled thrust chambers. The program consisted of firing two 5-pound thrust bipropellant rocket engines. Radiation cooled thrust chambers of molybdenum, coated with a Marquardt #RM-055 coating were used. This coating has been developed to withstand high temperature environments with high resistance to both oxidation and erosion. Test objectives were to evaluate both coating life and integrity in a liquid bipropellant rocket chamber environment. In addition to the contract effort, current Independent Research Corporate materials program efforts are reported, where the information usefully supplements the specific contracted effort.

Testing was completed at TMC Aero-Thermo Laboratory on the two combustion chambers, in a vacuum atmosphere. One chamber achieved a burn time of 130 seconds at a maximum recorded temperature of 2780°F and the second chamber accumulated 210 seconds of operation at a maximum recorded temperature of 3000°F. Both chambers burned through near the nozzle throat where very little coating was present to provide oxidation protection. The short life experienced in the test should be interpreted in reference to coating thickness and on this basis the coating still shows potential of achieving the long life expected of it.

Prior to the engine tests, two cylindrical molybdenum tubes coated with RM-055 were tested and achieved a life of 60 to 70 minutes without failure, respectively, at a temperature of 3100° F in a vacuum bell jar, indicating the potential capability of the coating at high temperature.



The conclusion that may be reached based on these tests is that further processing development is required in order to achieve a coating process procedure which can deposit the coating uniformally on the substrate material.

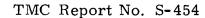
Additional work on RM-055 coating is being carried on with Marquardt Independent Research funds. The aim of the continuing program is still to learn how to utilize this modified silicon carbide coating as a high temperature coating for refractory metals. The silicon carbide coatings appear to be the only coating system which provides both resistance to high temperature environments as well as relative impermeability to the hydrogen diffusion that embrittles the refractory metals.

III. BACKGROUND

The development of the RM-055 coating system was undertaken for two specific reasons:

- A. Marquardt vapor deposition silicon carbide (RM-005) has demonstrated outstanding performance as a coating for graphite and tungsten substrates in high temperature rocket nozzle environments.
- B. If a silicon carbide material could be adapted to other refractory metal substrates such as molybdenum and tantalum-tungsten alloys, the operating temperatures of these alloys could be significantly increased. The maximum operating temperature is presently limited due to rapid oxidation of these refractory substrates.

The reason why pure silicon carbide is not practical as a coating for refractory metals other than tungsten is the low thermal expansion of SiC in relation to the metal substrates. However, if the composition could be modified sufficiently to make the coating expansion coefficient more compatible with the substrate without compromising the high temperature oxidation/erosion characteristics of silicon carbide, a superior coating for substrates such as molybdenum and tantalum - 10% tungsten would be possible.





The feasibility of modifying the composition and, consequently, the properties of silicon carbide for application to molybdenum and tantalum-10W substrates has been demonstrated in the laboratory at Marquardt. The new coating system, designated RM-055, has the same high hardness, dense structure, thermal shock resistance, and oxidation/erosion resistance as the pure silicon carbide coating. The modifications in the coating have resulted in a material which is more compatible with molybdenum from the standpoint of thermal expansion, and which is metallurgically bonded to the molybdenum substrate. These improvements make it possible to produce continuous, crack-free coatings with high thermal conductivity which will withstand repeated thermal cycles over a wide temperature range.

The coating is applied by vapor codeposition to all exposed surfaces to any desired thickness. A typical coated molybdenum test specimen is shown in Figure 1. The process for coating simple cylindrical specimens has become relatively routine, however, more study is needed to evaluate the effects of coating parameter variables on coating uniformity and composition and on the factors affecting the degree of bonding for small diameters and more complicated shapes.

Laboratory tests on coated specimens similar to that shown in Figure 1 demonstrated the compatibility of the modified coating systems with molybdenum substrates. Test samples subjected to repeated thermal cycling and thermal shock tests to temperatures of 3500°F in high velocity oxyacetylene gas stream showed no signs of deterioration or loss of protection. In addition, water quenching the coated cylinder from red heat failed to cause any cracking or spalling of the coating (See Figure 2). The specimens shown are made from sheet molybdenum which was rolled and seam welded. (Note the welding bead on the side of the specimens.) The coating integrity in the welded section is the same as in other areas.

The results of the initial laboratory tests conducted indicated that the coating has excellent oxidation resistance in a combustion environment at temperatures exceeding 3000°F.

Prior to this contract, all of the testing performed on the RM-055 coating system has consisted of laboratory tests. No demonstration testing in a rocket motor application was conducted. Therefore, the test firings of coated chambers and nozzles in the actual chemical and physical environment would help to determine the actual capabilities of the coating, to define where the problem areas are, and to indicate possible improvements in the system.





Figure 1



THERMAL SHOCK TEST ON RM-055 COATED MOLYBDENUM

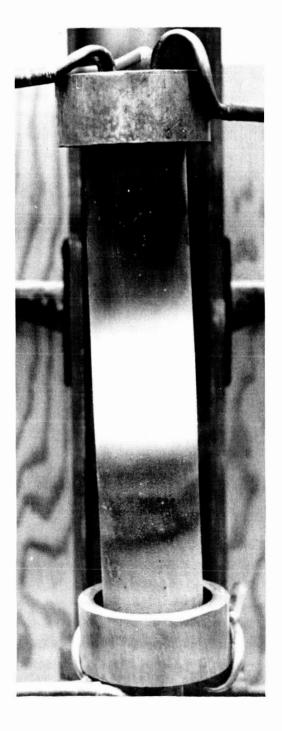


Figure 2

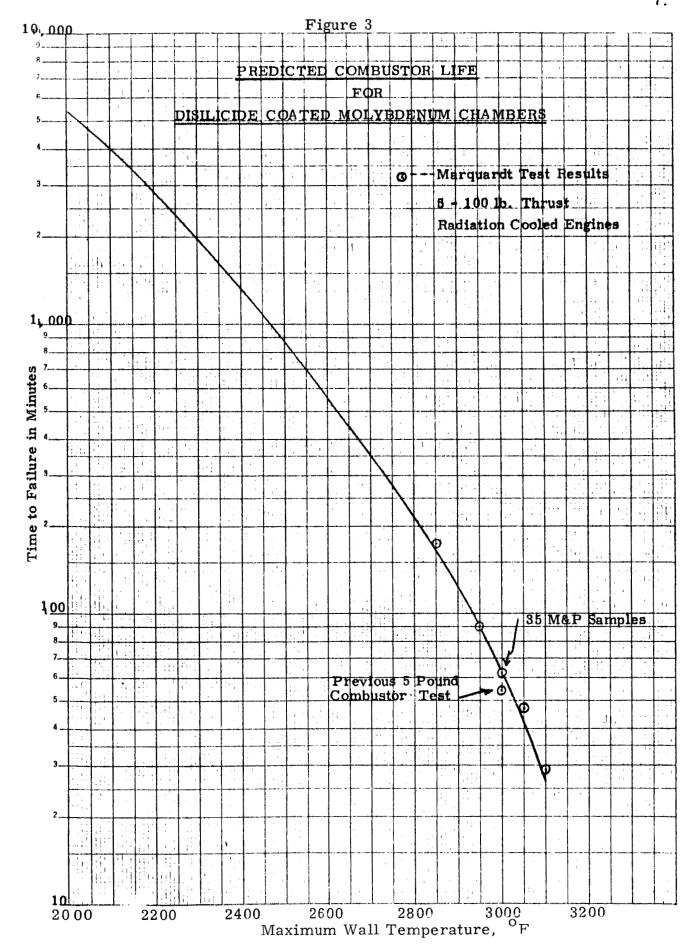
IV. PROGRAM APPROACH

The planned program approach was to conduct tests in the Marquardt Research Rocket Laboratory Pad E at sea level pressure conditions using N_2O_4 oxidizer and an aerozine-50 fuel. Two combustion chambers were to be tested to the same run schedule which consisted of:

- A. Trim runs as necessary to set the thrust at a value equivalent to 5-pounds of thrust at altitude, at an O/F of 1.6.
- B. Short steady state runs to check the maximum combustion chamber wall temperature. The expected temperature at this condition is 3100° F. If this temperature was not achieved, the flow rate and pressure are increased during additional trim runs until this condition is established.
- C. A steady state firing is then conducted for 3600 seconds or until failure whichever occurs first. The maximum allowable wall temperature during the steady state run is 3300°F.

The results of this test would be compared to TMC experience on dicilicide coated molybdenum chamber life, as shown on Figure 3. It is to be noted that the previous 5-pound disilicide coated molybdenum chamber tested achieved an equivalent life of 55 minutes to failure at 3000°F which showed good correlation with 35 material samples, which were torch tested at 3000°F and achieved a life equal to 62 minutes prior to failure.





V. DISCUSSION

A. Description of Hardware

The hardware utilized for the material evaluation consists of a 5-pound thrust bipropellant rocket engine as shown in Figure 4.

The injector configuration is a single doublet fixed orifice design. The fuel orifice is .0165 inches in diameter and the oxidizer orifice is .0195 inches in diameter.

Delivery of the propellants to the injector face is through two thin wall stainless steel tubes that are brazed to the injector face plate and protrudes slightly beyond it into the combustion chamber. These tubes have minimum heat capacity and high thermal resistance to heat flux from the injector face to the valve assembly.

The injector head is a stainless steel (series 321) fabricated for minimum weight and relatively low heat capacity.

The head contains a propellant channel adjacent to the seal groove between the injector and the chamber. The flow of fuel through this channel acts to keep the seal cool during steady state operation and during the thermal soak back after the engine has been pulsed. A unique Marquardt head to chamber joint reduces heat soak back to the injector head and provides for a live spring arrangement that maintains continuous loading at a fairly constant level for the all metallic seal. The injector head also provides the mounting surface for the combustion chamber and injector solenoid valves.

The engine incorporates two propellant control solenoid valves, one for oxidizer injection control and one for fuel injection control. The two solenoid actuated valves are identical and consist of an integral poppet valve and solenoid driver. High confidence in the reliability of this type of valve has been established at Marquardt by tests involving 10 million actuations of a single valve. The propellant control valves are of the poppet type with a conical seal of teflon and metal. This type of seat has proven itself in the Apollo and Advent programs and is capable of less than 2 cubic inches/year leakage rate after 1 million cycles. The actuator is of the single coil coaxial solenoid type. High response is achieved by minimizing the weight of the moving parts of the valve and by proper coil design.

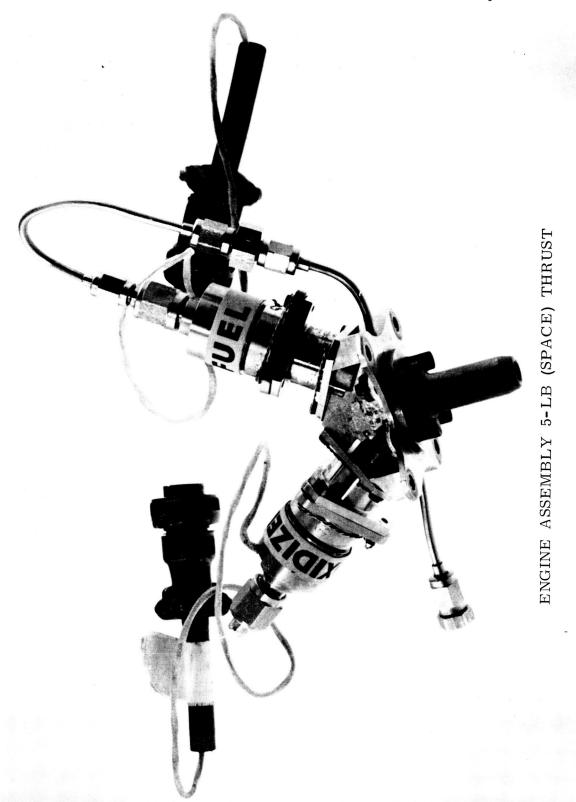


Figure 4



Figure 5 presents details of the solenoid valve design. Integral fixed orifices are incorporated in each propellant control solenoid valve to eliminate variations in required propellant flow rates resulting from variations in propellant supply line length and consequent pressure drop.

The combustion chamber used is a unique design developed during a TMC IR&D parameter research study. The chamber is characterized by its very short length made possible by the use of a premix chamber. This chamber with an L_{\ast} of 5 replaces a chamber previously used which had an L_{\ast} of 13, and has equivalent performance. The basic characteristics of the chamber are:

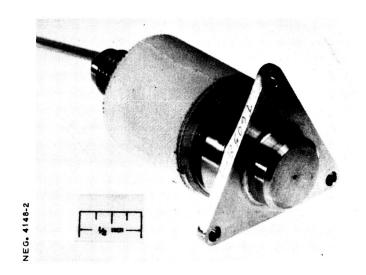
Design Chamber Pressure	=	90 psia
Nozzle Expansion Ratio	=	40/1 (at altitude) 1.65 (at S.L.)
A_c/A_T	=	4.2
L	=	1.26 inches
$D_{f T}$	=	.196 inches
$\rm L_{*}$	=	5
$D_{\mathbf{c}}$	=	.398 inches
t _{wall}	=	. 040 inches
Design Operating Temp.	=	$3000^{\mathrm{O}}\mathrm{F}$

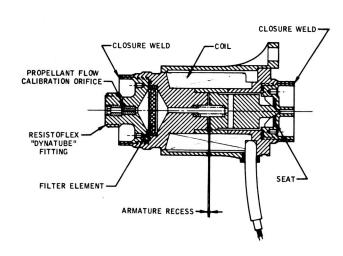
The chamber is constructed of molybdenum and is normally coated with a disilicide coating which was replaced in this program with the RM-055 silicon carbide coating. Figure 6 is a photo of the RM-055 sea level chamber configuration utilized.

Appendix B contains drawings of the engine assembly, the injector, and the combustion chamber as required by the contract.



SOLENOID INJECTOR VALVE 5 LB THRUST ROCKET ENGINE





Engine Thrust Level

Valve Weight (including filter)

Materials: Coil

Non-Magnetic

Magnetic

Potting & Insulation

Valve Type

Opening Force

Closing Force

Operating Pressure

Flow Rate (water)

Pressure Differential

Operating Voltage

Operating Current, Average

Operating Temperature

Opening Response at 24 volts D.C.

Closing Response at 24 volts D.C.

5 lbs.

0.31 lb.

Copper Wire

304L CRES

446 CRES

Ероху

Coaxial Flow, Poppet

Magnetomotive

Helical Spring

300 psig

40 pph

10 psi

19-29 volts D.C.

0.62 amperes

-80°F to +150°F

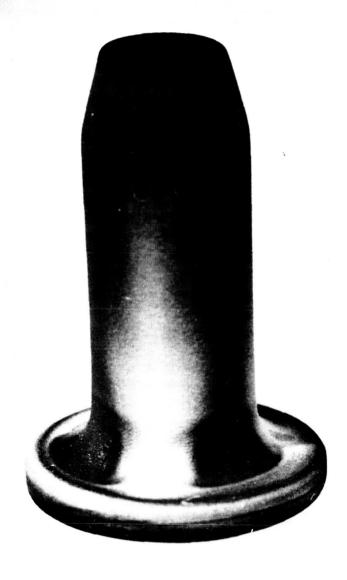
.008 seconds

.006 seconds

Figure 5



CHAMBER, 5-LB (SPACE) THRUST MOLYBDENUM - RM 055



NEG. 6236-2

B. Laboratory Effort

As described previously, the test plan formulated was to conduct Rocket Firing Tests on the chambers at 3100°F until failure or 3600 seconds are achieved and then compare these data with data on dicilicide coated chambers to determine relative life.

In order to make the temperature measurements with optical instruments, small cylindrical tubes of molybdenum and 90 Ta/10W were coated with RM-055 coating and calibrated in our laboratory.

A two color pyrometer (thermoscope) to be used in the test was calibrated using the coated molybdenum tube with a small hole in it. The tube is resistance heated in a bell jar to the calibration temperatures and the temperatures read by the thermoscope are recorded (See Figure 7).

Simultaneously a standard pyrometer records the True Temperature it sees looking at the black body hole in the tube samples. Using the black body measurement as a reference a final curve of Temperature vs. Millivolts is plotted.

The standard pyrometer in turn has been previously calibrated against a GE T24 pyrometer calibration lamp which has been certified by the Bureau of Standards. Emissivity values for the RM-055 coated tube are also computed during the process.

The initial calibrations conducted were at sea level conditions to simulate the planned test procedure, and under these conditions several tubes of molybdenum and 90 Ta/10W coated with RM-055 were heated through a range of temperatures up to 3100°F.

These tests indicated substantial changes in the coating coloration (formation of oxides) and a layering or scale formation which would make measurement of temperature by an optical pyrometer or thermoscope inaccurate. In addition, the degree of coating deterioration varied widely over the surface of the tube suggesting a variation of chemical composition of the coating deposited on the tube. Attempts at trying to utilize thermocouples on the chamber located under the coating, for temperature measurement were also unsuccessful due to chemical reactions between the couples and the coating at the operating temperatures.

Figure 7

INSTRUMENTATION

To verify if the coating on the chambers (coated in a previous batch) suffered the same defect, a 90 Ta/10W chamber was run in the ATL facility. It behaved in a similar manner. The external surface showed a heavy whitish scaly layer over the substrate and remaining coating. Rapid spalling and coating deterioration occurred.

The 90 Ta/10W chamber and the tubes which had been tested were sectioned longitudinally and then cross sections were taken and the parts examined metallographically. The coating on the outside surface of the 90 Ta/10W combustion chamber varied from 10 to 17 mills in thickness (See Figure 8). Some spalling of the oxidized coating was observed. The coating on the inside of the chamber was much thinner. Just downstream of the combustion chamber insert the coating was 3 to 4 mills thick (See Figure 9). Near the exit nozzle, the coating was less than 1/2 mill thick and was either burned or eroded away, thus exposing the Ta-10W substrate to excessive oxidation. Layer growth and cracking was noted in all of the coating.

The cracking of the coating and general oxidized appearance was also observed on the two emittance tubes examined. The only difference noted between the coatings on the 90~Ta/10W and molybdenum tubes was the appearance of melted oxide scale on certain areas of the 90~Ta/10W specimens (See Figures 10 and 11) but not on molybdenum.

Further metallographic and chemical analysis of the melted area indicated that the melted material was a low melting point oxide eutectic created when the oxidized tantalum substrate (Ta $_2$ O $_5$) flowed over the high melting point TiO $_2$ which is formed in the RM-055 coating. This lower temperature eutectic resulted in a more rapid removal of coating.

After the completion of the metallographic examination, samples were selected from the combustion chamber and submitted for chemical and X-ray analysis. The chemical analysis of the chamber coating indicated there was insufficient TiB in the modified SiC coating structure to obtain a proper match of coating and substrate expansion. X-ray diffraction patterns indicated a high titanium metal content. Substantial amounts of TiO₂ were found throught the coating with only small amounts of SiC being detected.

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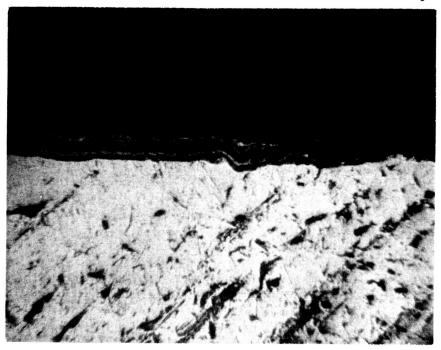


Figure 9 - Inside surface of Combustion Chamber Thin Impure Coating (3 mills) -300X-

NEG. C6481-5

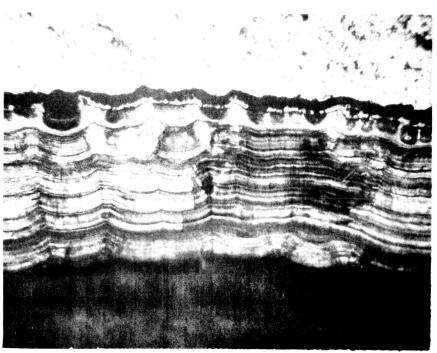
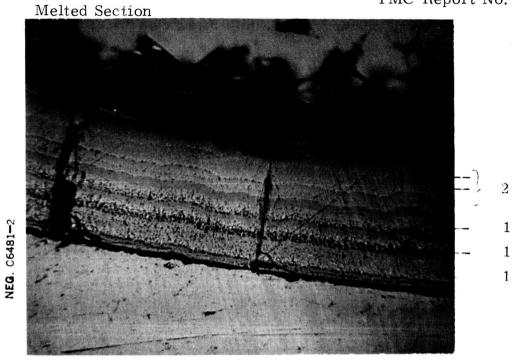


Figure 8 - Outside Combustion Chamber Good Coating (17 mills)
-100X-

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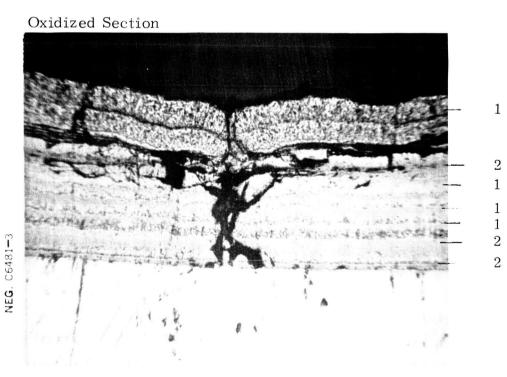


Figure 10 90 Ta 10W Tube 1 - SiC 2 - SiC-Ti-B Nonuniform Coating



Moly Tube

- 1 SiC Layer
- 2 Boron Rich Layer
- 3 Graphite Rich Layer

Figure 11

The SiC-TiB composition did not deposit uniformly. Cracking occurred due to the coefficient expansion of the metals.

18,



The conclusions reached as a result of this analysis indicated that the cracking and spalling of the coating were caused by an unbalanced deposition of the coating materials which resulted in differences in expansion characteristics of the coating and the substrate. The coating was deficient in boron thus containing a fair amount of free titanium metal. The oxidation of this titanium rich coating resulted in the formation of large amounts of TiO₂ scale. Based on these results, it was felt that tests of the molybdenum chambers with this poor coating as called out in the contract would not provide a valid test of RM-055 coating capability and that prior to conducting such tests, changes should be made in the coating composition and the processing procedures to obtain a more uniform thickness of coating on the chamber

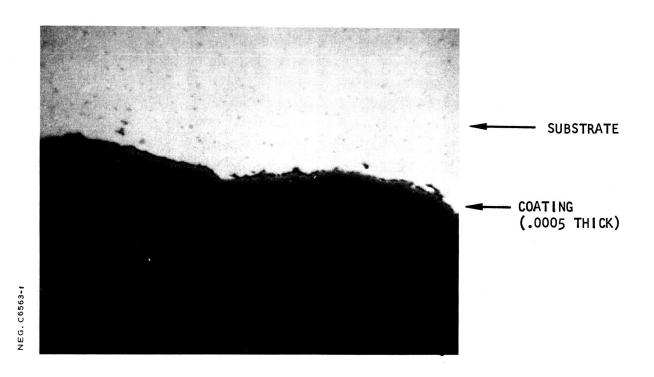
Additional tube samples were tested in the bell jar at sea level conditions and then at altitude conditions while changes in the coating parameters were made to eliminate the presence of free titanium and titanium oxide which had been present in the previous test results on the combustion chamber and the tube samples. These samples were then analyzed to measure the balance of the SiC and TiB mixture.

Two satisfactory molybdenum tube samples which were coated on the inside and outside with the RM-055 coating were subjected to testing at vacuum conditions in the bell jar. These tubes operated for 60 and 70 minutes without coating failure at $3100^{\circ}F$. However, upon shutdown the coating on the outside of the tube carcked indicating that an expansion mismatch still existed between the coating on the outside of the tube and the substrate.

A photomicrograph of the coated inner surface of the tube than ran 60 minutes under these conditions is shown in Figure 12. Although the coating was thin, .0005 inch, no cracking or spalling was evident and there were no gross deviations from the standard RM-055 composition.

Based on the results of the tube samples and the infeasibility of conducting further IR&D studies during the contract period, the decision was made to coat the engine combustion chambers on the inside only and run the engine tests at altitude vacuum conditions. These changes would thereby minimize the thermal expansion problem after cool down and would allow accurate temperature measurement during the test.

5 POUND MATERIALS EVALUATION MOLYBDENUM TUBE SAMPLE



Photomicrograph of the inside surface of the molybdenum tube that accumulated 60 minutes of operation in a vacuum Bell Jar at 3100°F.

Figure 12

600 X

20.

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Additional molybdenum tubes uncoated on the outside were then installed in the bell jar and the jar evacuated to altitude vacuum conditions to obtain a thermoscope calibration on the uncoated surface. The thermoscope calibration was conducted with the results shown in Figure 13. Emissivities for the uncoated moly are shown in Figure 14.

The two combustion chambers to be evaluated were stripped of their previous coating and then recoated on the inside only with RM-055. To insure that the coating thickness on these chambers would approach the .010 inch desired, a special support was constructed to force the reactant gasses to enter the small chamber hole at the flanged end and exit at the nozzle end. The thickness of the coating was controlled as a function of temperature and time.

In spite of these changes in the vapor deposition rig, the coating deposited on the chambers in a nonuniform manner with a very thin coating (.00025 to .0005 inches) near the throat section, and a very thick coating (.050 to .060 inches) near the flanged end. Since the thin coating occurred at a location where burn through does not usually occur and since the problem of coating composition appeared to be solved, the decision was made to utilize these combustion chambers for tests in spite of the thinness of the coating in the throat area. It is to be noted that all of the laboratory effort described herein was conducted as part of TMC independent research and development effort.

C. Engine Tests

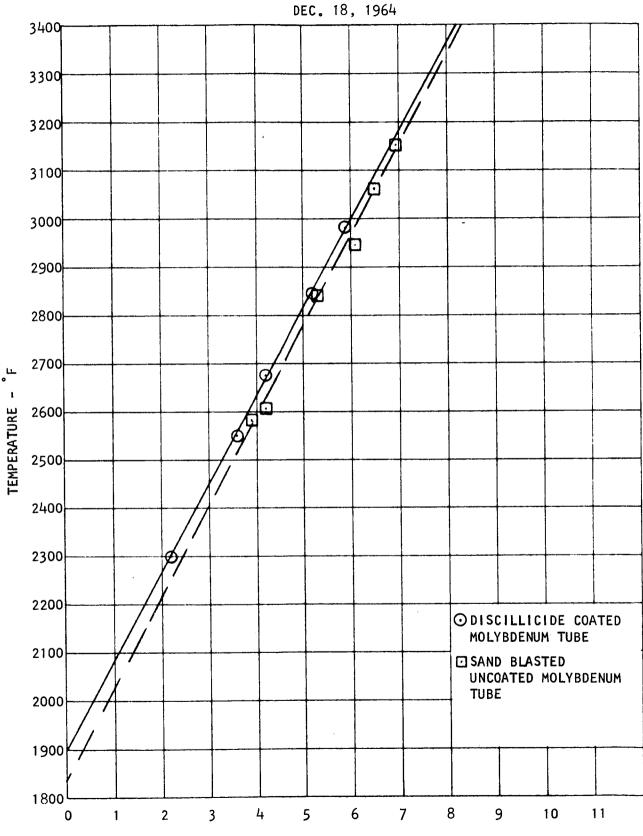
Based on the results of the laboratory effort, the decision was made to conduct the rocket engine firing tests at altitude conditions in TMC Aero-Thermo Laboratory facility. The instrumentation and recorders utilized and the parameters measured are as noted in Table I.



DATE_____

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THERMOSCOPE LABORATORY CALIBRATION



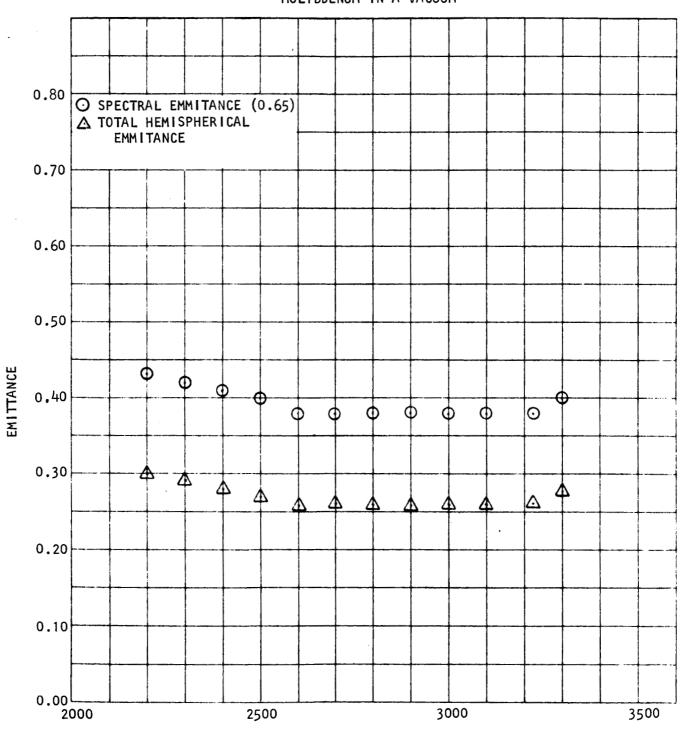
MILLIVOLTS

Figure 13 Page 22



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TOTAL HEMISPHERICAL AND SPECTRAL EMITTANCE VS. TEMPERATURE MOLYBDENUM IN A VACUUM



TEMPERATURE - °F

z
NOI
\mathcal{L}
II
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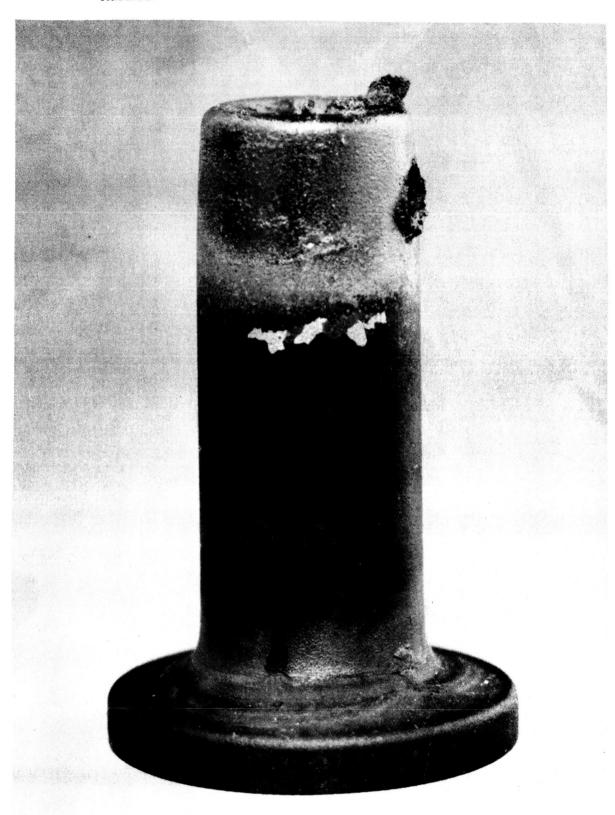
	Recorder	Bristol and Oscillograph	Visual and Oscillograph	Visual and Oscillograph	Digital Voltmeter, Visual and Oscillograph	Digital Voltmeter, Visual and Oscillograph	Bristol and Oscillograph	Recorder	Visual, Bristol and Oscillograph	Visual, Bristol and Oscillograph	Recorder	Bristol and Oscillograph	Recorder	Brown	Bristol	Brown	Bristol	Bristol	
- INSTRUMENTATION	Type of Instrument	Taber Transducer (Strain Gage)				₽	C. E. C. Absolute Pressure Transducer	Type of Instrument	Cox Turbine Type Flowmeter	Cox Turbine Type Flowmeter	Type of Instrument	Lebow Load Cell (Strain Gage Semiconductor)	Type of Instrument	Thermocouples			->	<pre>2 color Pyrometer (''Thermoscope'')</pre>	
TABLE	Range	0-1 00 psia	0-300 psig	0-300 psig	0-300 psig	0-300 psig	020 psia	Range	0008 pps	0 012 pps	Range	spunod 9-0	Range	0-100 ⁰ F	0-300°F	$0\text{-}100^{ m OF}$	0-300 ⁰ F	0-3500 ⁰ F	
	Pressures	$ extsf{P}_{ extsf{c}}$ Thrust Chamber	$ extsf{P}_{ extsf{Fs}}$ Fuel Supply	P _{oxs} Oxidizer Supply	$ m P_{FM}$ Fuel Manifold	$ m P_{oxM}$ Oxidizer Manifold	$_{ m O}^{}$ Cell Pressure	Flows	$ m W_{ m F}$ Fuel Flow Rate	W _{ox} Oxidizer Flow Rate	Thrust	$^{ m F}_{ m Test}$ Thrust	Temperatures	${ m T}_{ m F}$ Fuel Supply	$ extsf{T}_{ extsf{FV}}$ Fuel Valve	T Oxidizer Supply	Toxy Oxidizer Valve	Twall Combustion Chamber Outer Wall	

The propellants utilized were (50% UDMH/50% $\rm N_2H_4$) for the fuel and $\rm N_2O_4$ for the oxidizer. The first configuration to be tested was engine assembly T8012 combustion chamber P/N T8013 S/N 005. Hot firing trim runs at altitude were conducted to achieve the required test condition of a combustion chamber wall temperature of 3100°F. A total of 10 hot firing trim runs were conducted in which the chamber was subjected to 130 seconds of operation at a maximum recorded wall temperature of 2780°F. The trim runs were stopped due to a burn through of the combustion chamber in the throat area.

The second chamber tested was engine assembly T8012 combustion chamber P/N 9024-501 S/N 002. A total of 16 hot firing trim runs were conducted on this chamber during which it accumulated 210 seconds of operation at a maximum recorded temperature of $3000^{\circ}\mathrm{F}$. Testing was terminated due to a combustion chamber burn through near the throat area of the chamber.

Figures 15 through 24 show the condition of the combustion chambers at the completion of the test.

The failures occurred at the location where the coating was extremely thin. Measurements made before the test indicate .00025 inches average thickness on one chamber and .0005 inches on the other. Target thickness for the coating was .010 inches. Reduced and representative plotted data plus data summary sheets on all burn runs are presented and described in Appendix A.



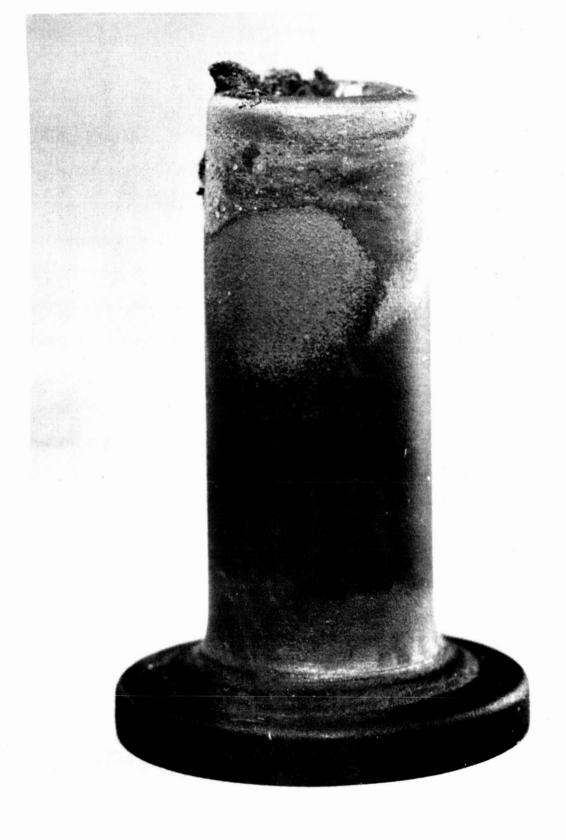
MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T8013 S/N 005 BURN TIME--130 SECONDS ATL-PAD E LOCATION--UNDER FUEL VALVE (U) 19 DEC 64 TEST 3237

Figure 15



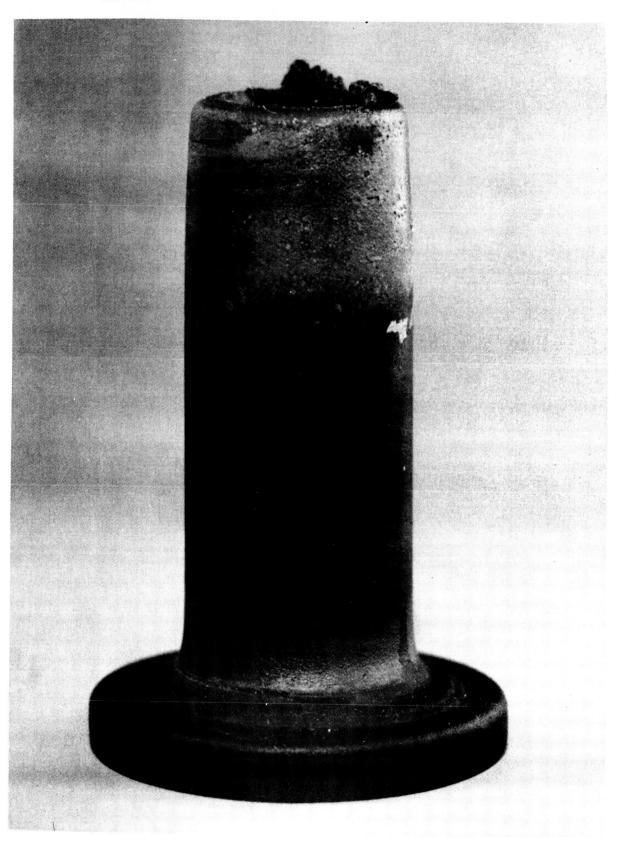
ATL-PAD E 19 DEC 64 MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T8013 S/N 005 BURN TIME -- 130 SECONDS LOCATION--90° CLOCKWISE FROM FUEL VALVE (U) TEST 3237

Figure 16



ATL-PAD E 19 DEC 64 MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T8013 S/N OO5 BURN TIME--130 SECONDS LOCATION--180° CLOCKWISE FROM FUEL VALVE (U) TEST 3237

Figure 17



ATL-PAD E 19 DEC 64 MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T8013 S/N OC5 BURN TIME--130 SECONDS ATL-PADE LOCATION--270° CLOCKWISE FROM FUEL VALVE (U) TEST 3237

Figure 18



ATL-PAD E 19 DEC 64 MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T8013 S/N 005 BURN TIME--130 SECONDS
LOCATION--END VIEW (U)
TEST 3237

Figure 19



FI-MOLY COMBUSTION CHAMBER - RMO55 COATING BURN TIME -- 240 SECONDS

Figure 20



MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T9024-501 S/N 002 BURN TIME--240 SECONDS
LOCATION--90° CLOCKWISE FROM FUEL VALVE (U)
22 DEC 64

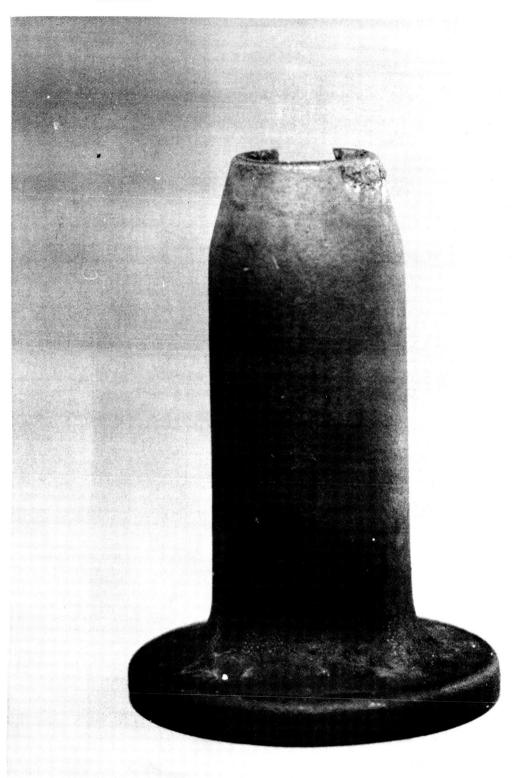
Figure 21



MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T9024-501 S/N OO2 BURN TIME--240 SECONDS LOCATION--180° CLOCKWISE FROM FUEL VALVE (U) ATL-PAD E TEST 3237

Figure 22

NEG. T3237-15



MATERIALS EVALUATION .5 TI-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T9024-501 S/N OO2 BURN TIME--240 SECONDS
LOCATION--270° CLOCKWISE FROM FUEL VALVE (u)
22 DEC 64

Figure 23

NEG. T3237-16

MATERIALS EVALUATION .5 Ti-MOLY COMBUSTION CHAMBER - RMO55 COATING P/N T9024-501 S/N COZ BURN TIME--240 SECONDS ATL-PAD E LOCATION--END VIEW (U) 22 DEC 64 TEST 3237

Figure 24

D. Failure Analysis and IR&D Effort

The fired combustion chambers were sectioned longitudinally. One half of each chamber was polished and etched and the other half was cut into four transverse sections. Chemical and X-ray diffraction samples were taken from these sections and submitted for analysis. The other sections were then mounted, ground, and polished for metallographic analysis.

Photomicrographs of each of the chambers plus photographs of the longitudinal section of the chambers are presented in Figures 25 through 30. The coating thickness on the inner surface of both chambers varied widely along the length. On the insert, the coating was about .001 inch thick. From the insert on toward the throat the coating thickness varied from .061 inch on one chamber and .052 inch on the other chamber to nothing at the throat area (See Figures 27 and 30). Oxidation and erosion of the substrate was noted in the throat area of both chambers where there was no coating present.

The transverse cross sections of both chambers just upstream of the throat showed coating thickness of .007 inches and .005 inches, respectively, and showed that the coating in these areas effectively protected the substrate. Figure 29 shows the throat area failure on chamber T8013 and clearly illustrates the oxidized and eroded area.

Figure 28 shows a typical thermal stress crack which occurred in the coating in an area where the thickness was .023 inches. In the area of the chamber where the coating was extremely thick .050 - .060 inches, many thermal stress cracks were observed.

Results of the chemical analysis for silicon, boron, and titanium showed no gross deviations from the standard RM-055 chemical composition. X-ray diffraction results showed only SiC and TiB present with no indication of free titanium metal, thus indicating that the composition problems occurring earlier in the program as described under the laboratory effort had been eliminated.

Based on the fact that the coating in the throat area of the chambers was initially extremely thin before testing, the early failure was attributed to erosion of the thin layer and exposure of the molybdenum structure to the hot exhaust products resulting in the observed burn through in the throat area.

Molybdenum Chamber - RM-055 Coating P/N T8013 S/N 005 Burn Time 130 seconds Longitudinal Cross Section

NEG. 6563-10

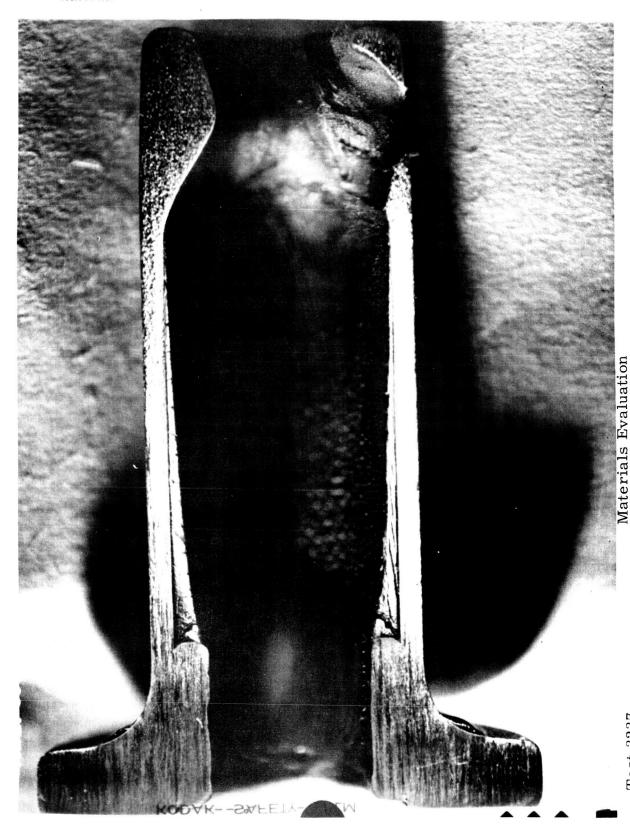


Figure 25

ATL Pad E 19 December 1964

Test 3237

NEG. 6563-9

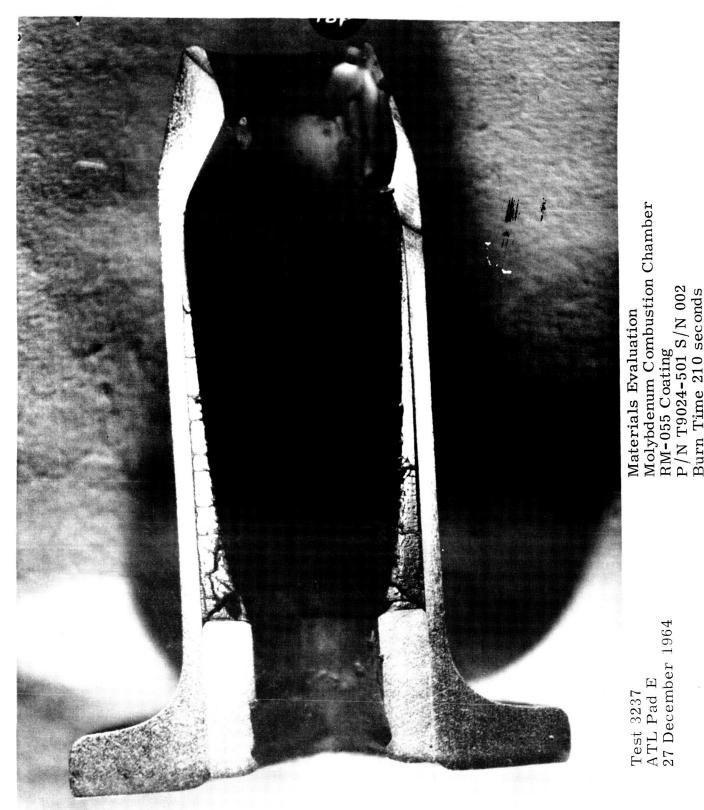
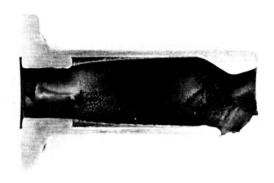


Figure 26

Test 3237 ATL Pad E 27 December 1964

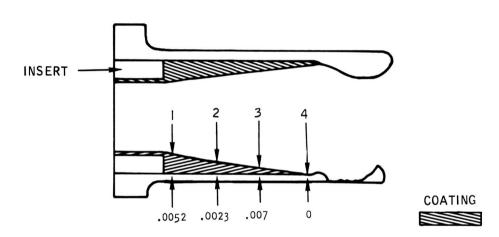
38.

TEST 3237 ATL PAD E P/N T8013 S/N 005 BURN TIME 130 SECONDS

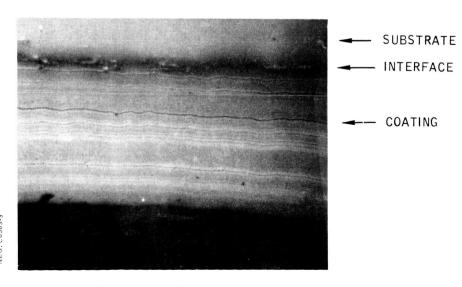


56 **2**8 16 24 32 40 48 56 **3**

2X



COATING THICKNESS IN INCHES

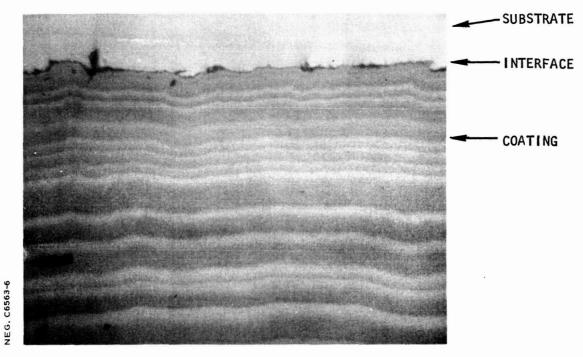


LOCATION 3 - CROSS SECTION COATING THICKNESS - .007 INCH UNIFORM COATING COMPOSITION 300 X

TEST 3237 ATL PAD E P/N T8013 S/N 005 DEC. 19, 1964

BURN TIME 130 SECONDS PHOTOMICROGRAPHS

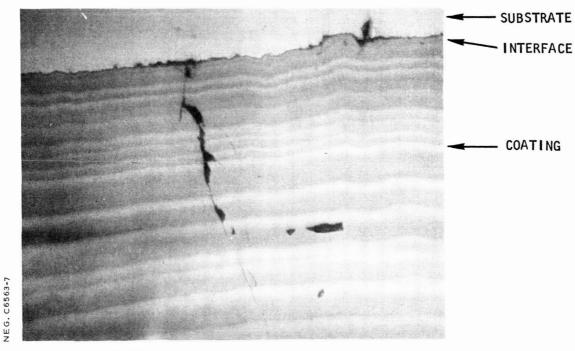
TMC Report No. S-454 Figure 28 Page 40



REF. FIG. - LOCATION 2 CROSS SECTION

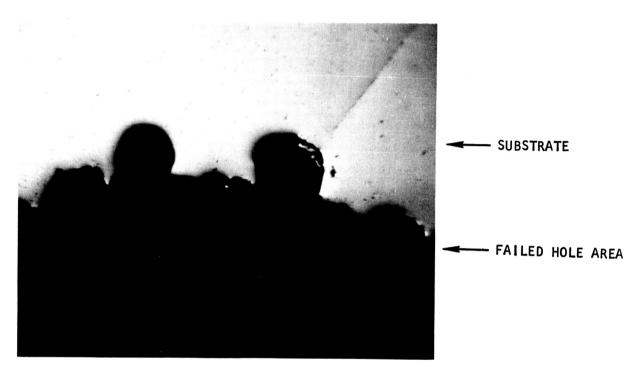
COATING THICKNESS 0.0023 INCH

300X



REF. FIG. - LOCATION 2 CROSS SECTION COATING THICHNESS 0.0023 INCH THERMAL CRACK 300X

TEST 3237 ATL PAD E P/N T8013 S/N 005 BURN TIME 130 SECONDS PHOTOMICROGRAPH



REF. FIGURE 27 LOCATION FAILED HOLE AREA BELOW 4. NO COATING LEFT TO PROTECT SUBSTRATE.

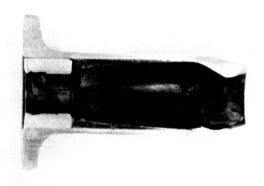
300 X

TMC Report No. S-454

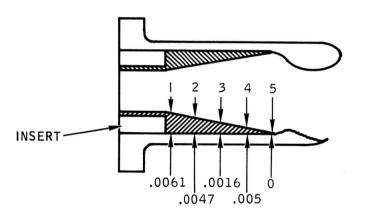
Figure 29

41.

TEST 3237 ATL PAD E P/N 9024-501 S/N 002 DEC 22, 1964 BURN TIME 210 SECONDS PHOTOMICROGRAPH

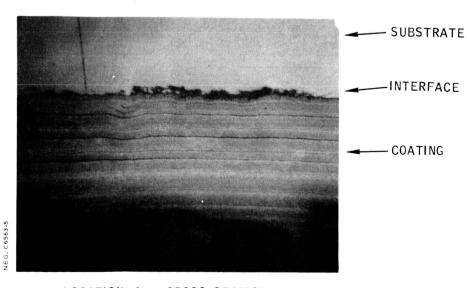




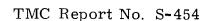


COATING

COATING THICKNESS IN INCHES



LOCATION 4 - CROSS SECTION
COATING THICKNESS - .005 INCHES
UNIFORM COATING COMPOSITION
300 X





In spite of the early failure with the rocket chamber, the test results offer some reasons for optimism in regard to the coating. The optimism arises from a possible interpretation of the results. If the assumption is made that the coating life is proportional to the thickness (i.e., the chemical or physical process occurs at a constant rate under the test conditions) the estimated life of the target .010 inch coating would be about 20 to 40 times that of the coating tested. This would bring the estimated life of a .010 inch coating to somewhere between 43 to 86 minutes. This simple extrapolation may be expected to predict shorter life than may occur for two reasons. One, the thin coating is more susceptible to small imperfections and defects than a thick coating and hence would give a faster erosion rate per unit thickness. Second, coating life appears to be nonlinear with thickness (i.e., the oxidation coating often provides a small amount of protection thereby providing a longer life than predicted from thin films).

The test data are disappointing because they reveal that substantial work still remains to define the vapor deposition process parameters and techniques which will produce uniformity of deposition. Observations made at Marquardt clearly indicate that the dimensions and shape of the chamber play an important role in respect to uniformity of deposit and susceptibility to cracking due to small mismatches of expansion coefficients. Coating of one inch diameter tubes appears more uniform than the 1/2 inch chambers. The RM-055 coating applied to simple six inch diameter advanced air breathing combustion chamber components did not seem to have the number or degree of the difficulties encountered with the small contoured rocket chamber. The complex shape and variable wall thickness affects the temperature distribution of the induction heated chamber in two ways. Since the deposition rate is affected by the chamber temperature, the material will deposit unevenly if temperature differences occur over the surface of the chamber. Furthermore, variation of the velocity of the deposition gases through the chamber also affects the deposition rate producing another cause for variations in the thickness of coating. The small radius of curvature of these chambers also appears to have an important effect on sensitivity of the coating to cracks due to mismatches of thermal expansion coefficients.

Successful application of RM-055 to larger and simpler configurations is also being experienced at The Marquardt Corporation for another application. Under another of the company's Independent Research programs, the RM-055 coating is also being investigated as a hydrogen barrier. The application is related to an ablative rocket chamber design shown in Figure 31. This chamber utilizes a graphite insert which is prestressed in compression by use of a 90 Ta/10W cylincrical sleeve to increase the load carrying capability of the graphite. The back side of the sleeve is exposed to silica phenolic ablative material. It is a design requirement that the 90 Ta/10W sleeve be protected from the ablative outgassing of hydrogen by a protective coating. Tests previously conducted by TMC indicated that the RM-055 coating is relatively impermeable to hydrogen gas diffusion and would make an excellent hydrogen barrier.

The requirement for this application is for a very thin flash coating on the cylindrical sleeve to act as a diffusion barrier. The engine design is such that the coating does not come in contact with any high velocity eroding gases so that a very thin coating can be utilized. 90 Ta/10W strips have been flash coated with a .0005 inch coating and 180° bend tests were performed without base material failure thereby indicating that hydrogen embrittlement did not occur. This work indicates the potential of this coating and explains in part our optimism that it has a significant place in high temperature materials technology.

COMPOSITE COMBUSTION CHAMBER ASSEMBLY

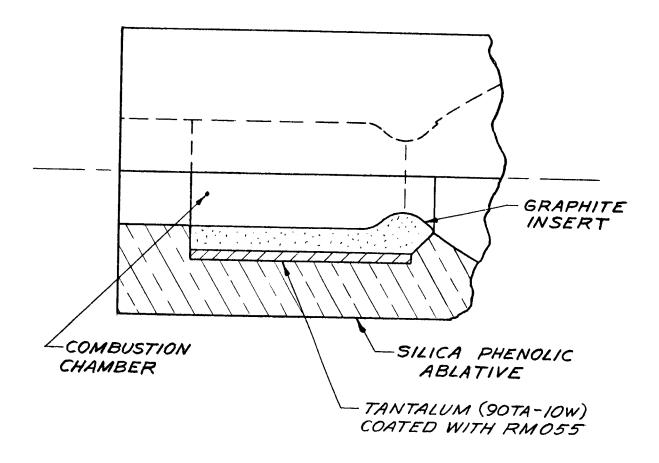


Figure 31

VI. CONCLUSIONS

- 1. The two combustion chambers tested failed in short periods of time due to a very thin coating that rapidly eroded away and exposed the molybdenum substrate to the hot exhaust products.
- 2. The short life experienced in the test should be interpreted in reference to coating thickness and on this basis the coating still shows potential of achieving the long life expected of it.
- 3. Additional processing development is required to define the vapor deposition process parameters and techniques which will produce a uniform thickness of the deposit on small contoured parts.
- 4. Independent research and development effort at Marquardt continues to indicate the promising potential of this coating system. The silicon carbide coating appears to be the only coating system which can provide the resistance to high temperature environments as well as being relatively impermeable to the hydrogen diffusion that embrittles the refractory metals.

APPENDIX A

DATA PRESENTATION

<u>A ND</u>

SUMMARY SHEETS

APPENDIX A

DATA PRESENTATION AND SUMMARY SHEETS

Presented herein is the reduced and plotted data plus the data summary sheets. The symbology and computations used are as noted below.

P = Cell pressure

P₁ = A cell pressure = to .0999 psia.
The actual test data was related to this condition so as to compare actual data with theoretical performance data which was available for this condition through a previous IBM performance summary.

P/N T8013 S/N 005	P/N T9024-501 S/N 002			
A_{T} = Throat area = $.0373 \text{ in}^2$	$A_{T} = .0387 in^2$			
Ae = Exit area = .0615	Ae = $.0633 \text{ in}^2$			
$Ae/A_T = 1.65$	$Ae/A_T = 1.64$			
F _{Test} = Thrust indicated				
F_1 = Thrust achieved at P_0 = .0999 psia = $F_{Test}^+(P_0 - P_1)Ae$				
Fvacuum Thrust that would have been achieved in a vacuum with a nozzle expansion ratio of 40/1				

$$W_F$$
 = Fuel flow

$$W_{OX}$$
 = Oxidizer flow

$$W_{ox}/W_{F} = Oxidizer/Fuel ratio$$

$$W_p$$
 = Total propellant flow = $W_F + W_{ox}$

 I_{sp1} = Specific impulse = F_1/W_p

Isp Theor 1 = Specific impulse (Theoretical) provided by IBM data for the O/F and chamber pressure utilized

 $I_{sp} \mathcal{N} = \text{Specific impulse efficiency} = \frac{I_{sp 1}}{I_{sp Theor}} \times 100$

I sp Vacuum = The specific impulse that would have been achieved in a space vacuum with a 40/1 nozzle. This value has been calculated using the I reduced by 2% to take care of additional nozzle losses.

I_{sp} Vacuum Theor = The theoretical vacuum impulse provided from IBM performance data.

P_c = Combustion chamber pressure

g = Gravitational constant = 32.2 ft/sec²

 $C_* = \frac{P_c A_T g}{W_p}$

C*Theoretical = Theoretical value of C* obtained from IBM performance data

 $C_* \sim C_* \text{ efficiency} = \frac{C_*}{C_* \text{Theoretical}}$

Several aspects of the data are worthy of discussion. First, the original combustion chamber design had the following dimensions after coating a chamber with its original disilicide coating.

 D_{T} = .196 inches

 $A_{T} = .0302 \text{ inches}^2$

 $A_{C}/A_{T} = .0302$

L_{*} = 5.0

D_c = .260 inches

Since the desired RM-055 coating thickness was .010 inches on a side or .020 inches on the diameter, the dimensions of the RM-055 coated chamber desired prior to coating were

 D_{T} = .216 inches

 $D_e = .280 \text{ inches}$

 $A_T = .0366 \text{ inches}^2$

When the very thin coating was deposited on the chambers in the throat area, this resulted in a larger throat area than anticipated and thereby a lower combustion chamber pressure for an equivalent thrust value as shown by the following calculations.

1. $_{
m T}$ Previous tests on disilicide coated chambers with an A $_{
m T}$ = .0302 in 2 produced 5-pounds of vacuum thrust at a chamber pressure of 91 psia

$$F = P_c A_T C_F$$
 $C_F = \frac{F}{P_c A_T} = \frac{5}{(91)(.0302)} = 1.82$

2. Assuming the same $C_{\overline{F}}$ an expected combustion chamber pressure can be calculated for the two chambers tested.

$$P/N$$
 T8013 S/N 005 -- A_{T} actual = .0373 inches

$$P_c = \frac{F}{C_F A_{T \text{ actual}}} = \frac{5}{(1.82)(.0373)} = 73.5 \text{ psia}$$

$$P/N T9024-501 S/N 002 A_{T actual} = .0387$$

$$P_c = \frac{F}{C_F A_{T \text{ actual}}} = \frac{5}{(1.82)(.0387)} = 71 \text{ psia}$$

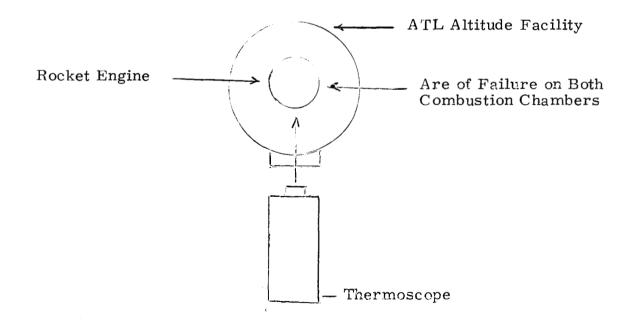
Since this was a materials evaluation test where the primary purpose of the engine is to create a temperature environment for the chamber, it was felt that this type of chamber pressure deviation was acceptable.

In addition to the change in throat dimensions, the large variations in coating deposition did change the interior contour, the L_{\star} , and expansion section which normally existed behind the chamber insert.

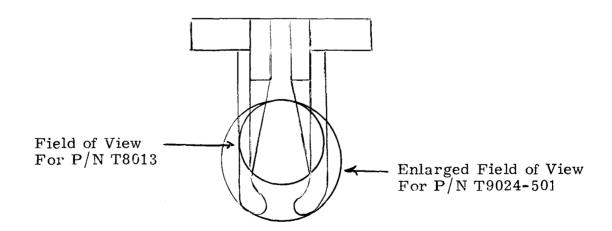
These discrepancies are pointed out to indicate that the performance data as presented does not necessarily correspond to the ideal drawing configuration but is applicable to only this test configuration.

The actual combustion chamber pressures achieved during the test correlate well with the calculations noted above.

Another parameter worthy of discussion is combustion chamber wall temperature. In utilizing the thermoscope for temperature measurement the thermoscope was focused at the plane of the combustion chamber in line with the fuel valve location where previous experience indicated burn through normally occurs. The actual failure on both combustion chambers occurred 90° from the plane of the thermoscope as noted below:



On P/N T8013 S/N 005, the field of view of the thermoscope was as shown below to cover the expected area where burn through might occur, based on previous tests on this type of chamber. When P/N T8013 failed in the throat area below the field view noted, the thermoscope field of view was increased to cover the lower portion of the chamber as indicated below.



Since the thermoscope optically averages the temperature in its field of view, it is believed this change in the field of view may have significance in reviewing the plotted data.

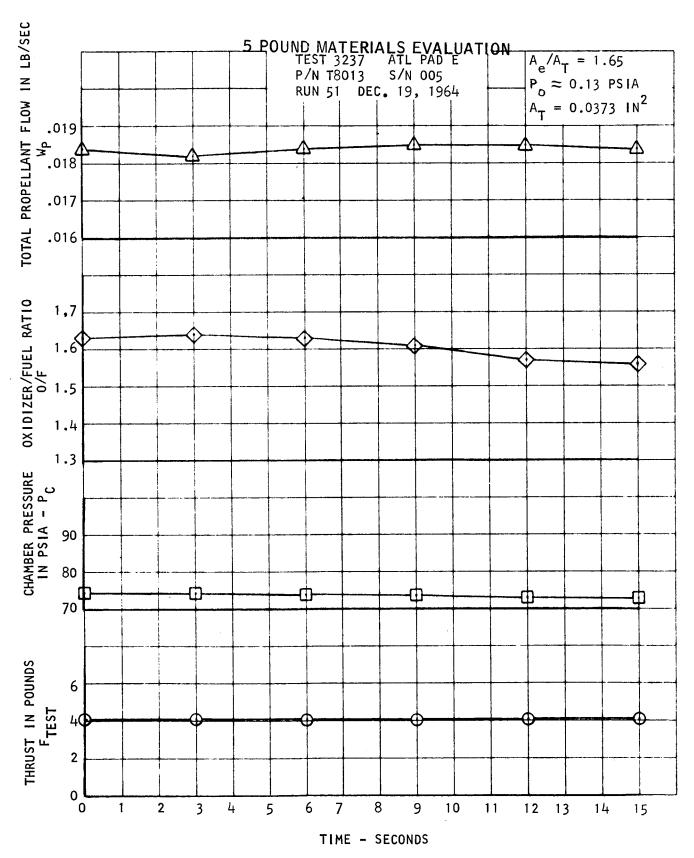
It is to be noted that the second combustion chamber recorded temperatures about 200° higher than the first chamber under similar operating conditions and this difference is believed to be partially due to the change in field of view such that the second chamber has recorded temperature values which are more representative of the true temperature in the area where burn through finally occurred.

Presented herein are representative plotted data from some of the Rocket Firings conducted during the test program. In addition, all reduced and calculated data are included. Plotted data of all Rocket Firing runs are on file at TMC for future reference.

REPRESENTATIVE PLOTTED DATA

P/N T8013 S/N 005







PREPARED	BY	***		
DATE			-	

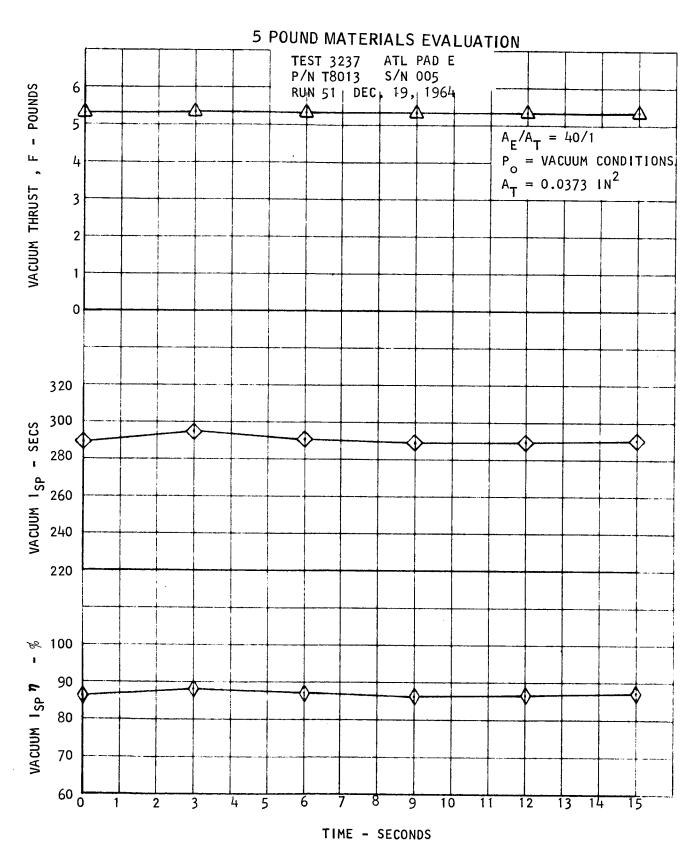


Figure 33 A-10



DATE_____

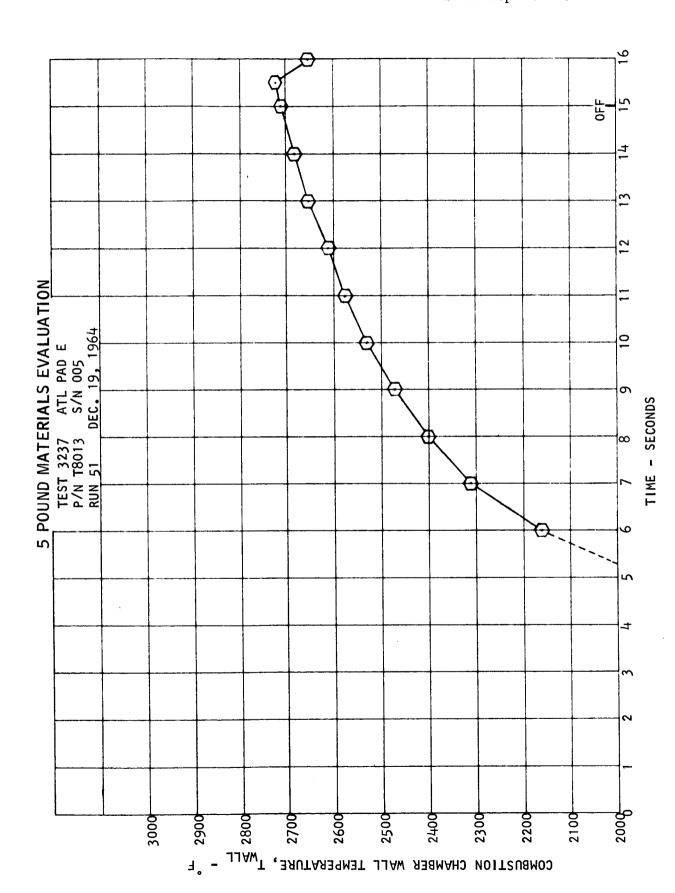


Figure 34 A-11

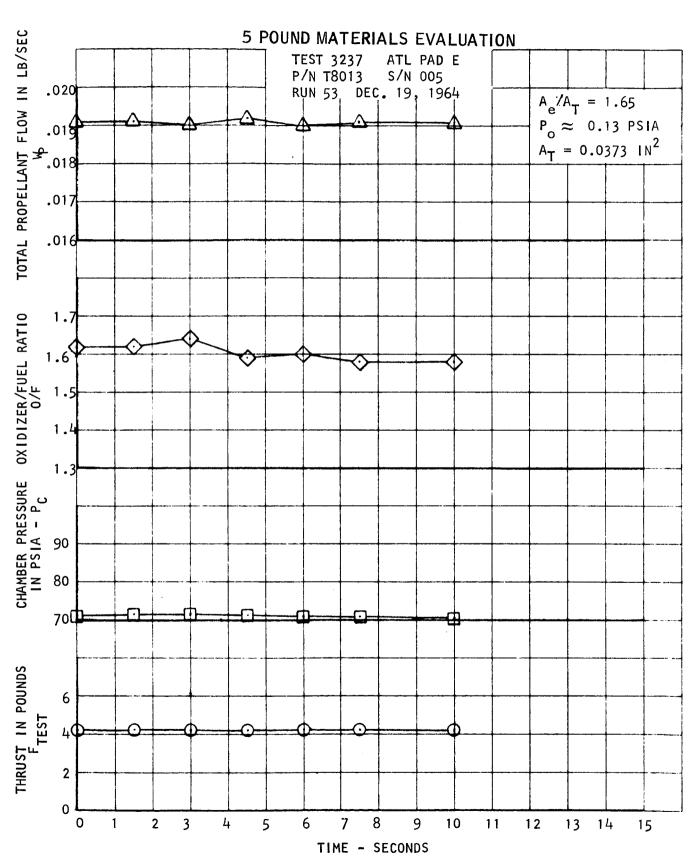


Figure 35 A-12

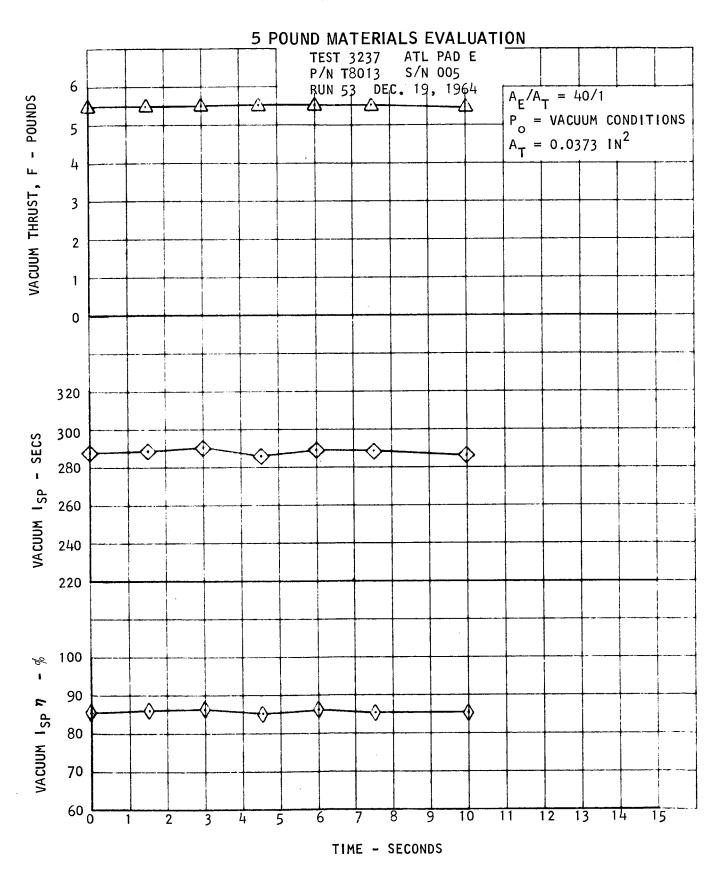


Figure 36 A-13

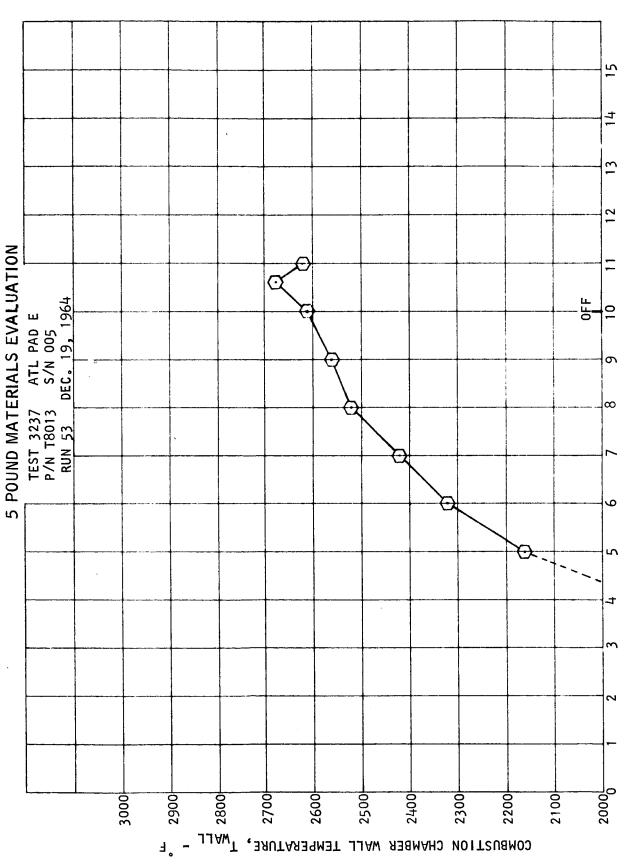
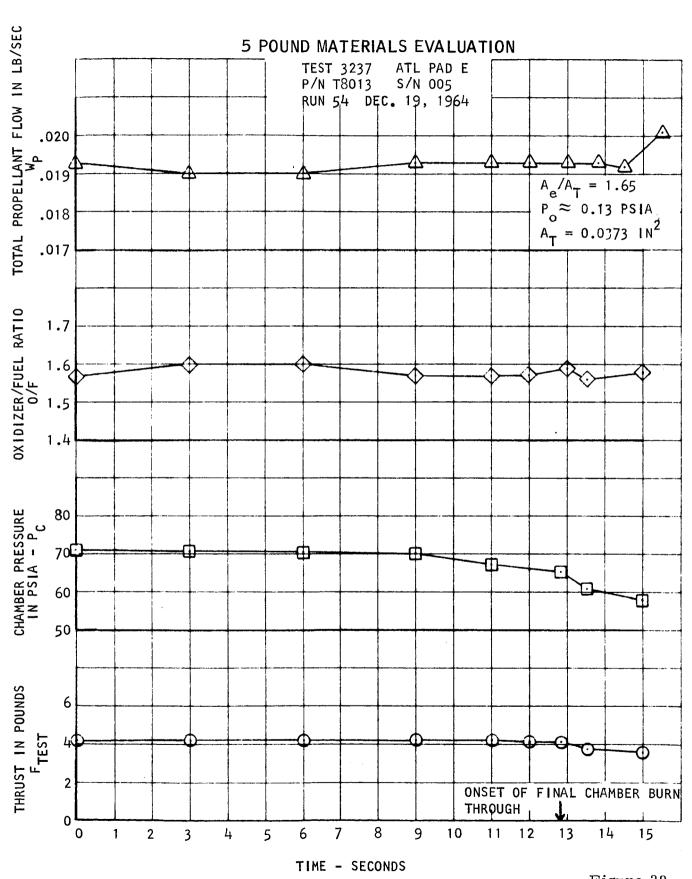


Figure 37 A-14

TIME - SECONDS





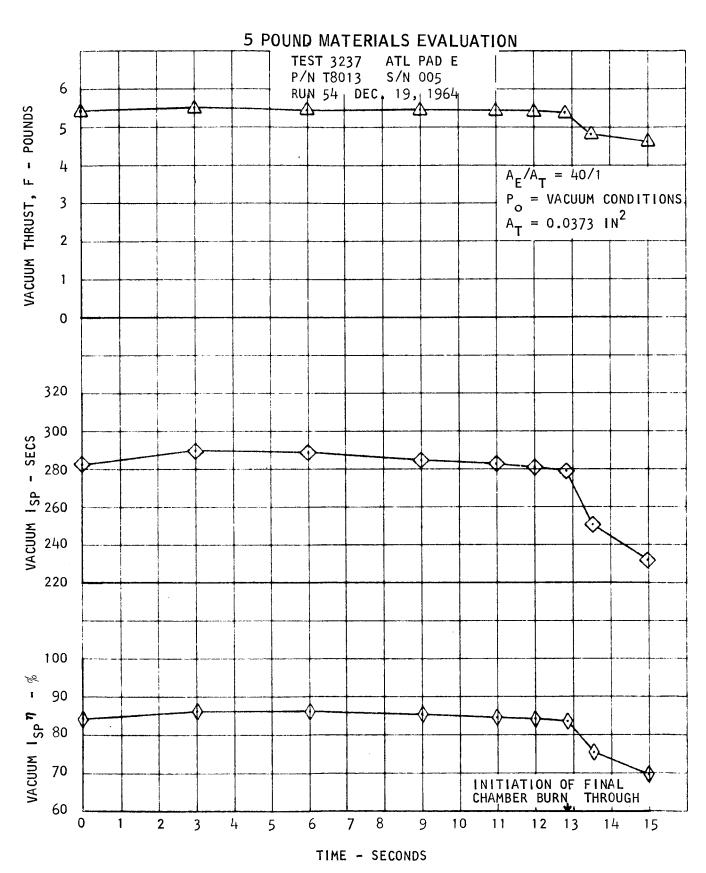
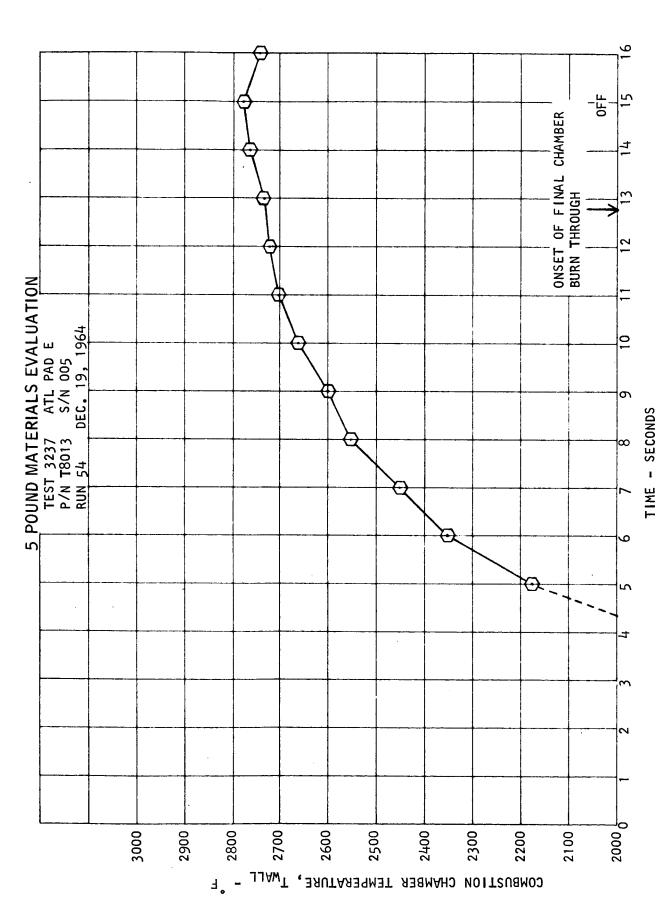


Figure 39 A-16



PREPARED BY_____

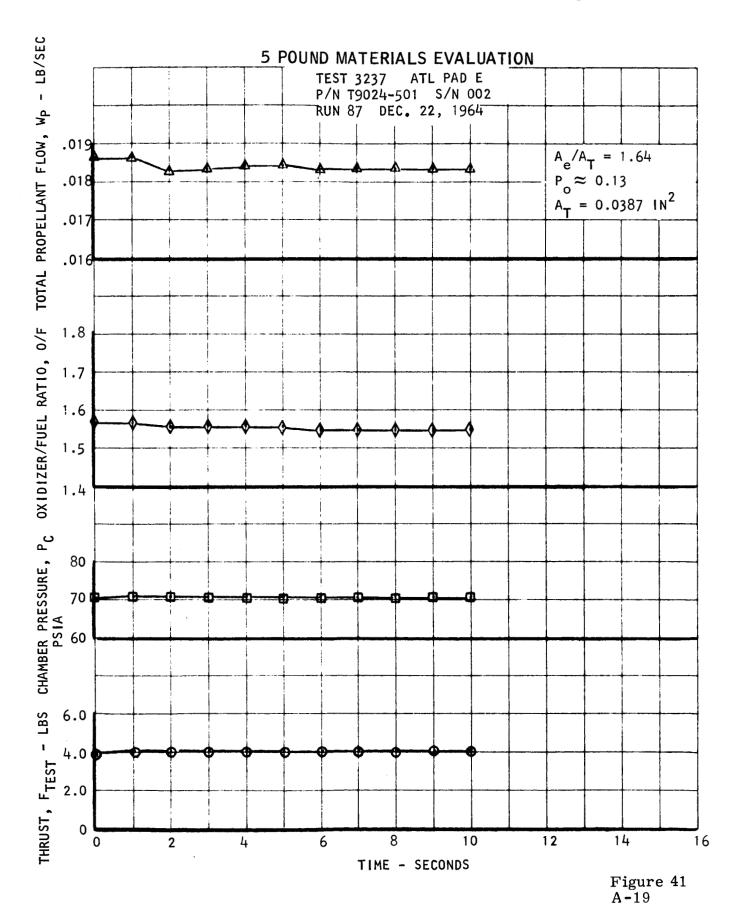


REPRESENTATIVE PLOTTED DATA

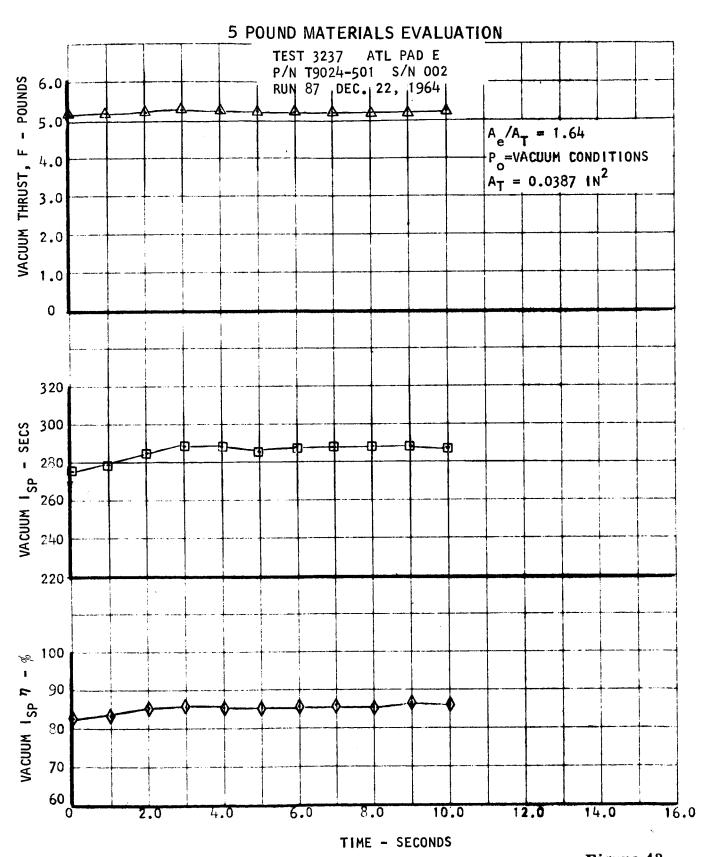
P/N T9024 - 501 S/N 002



DATE 1-25-65

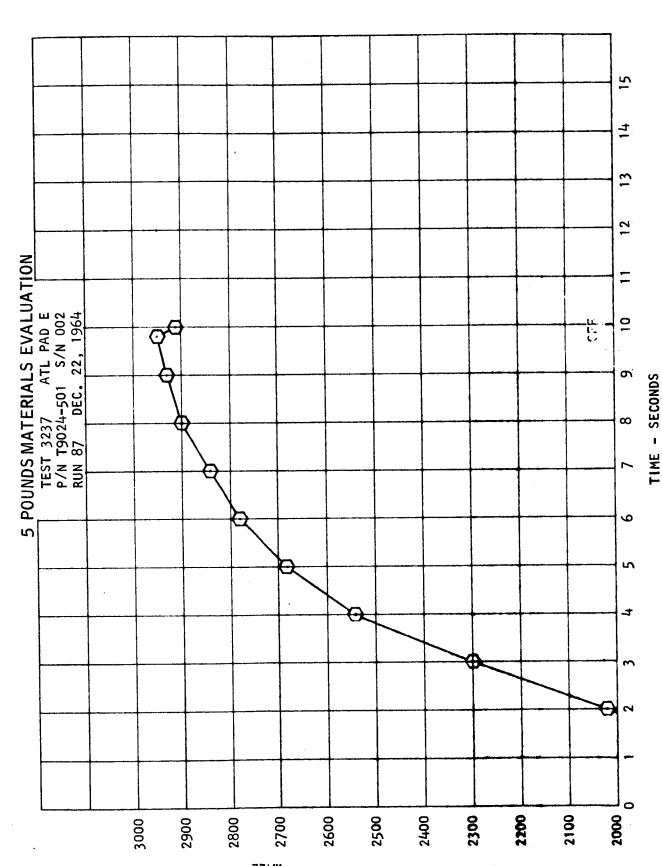






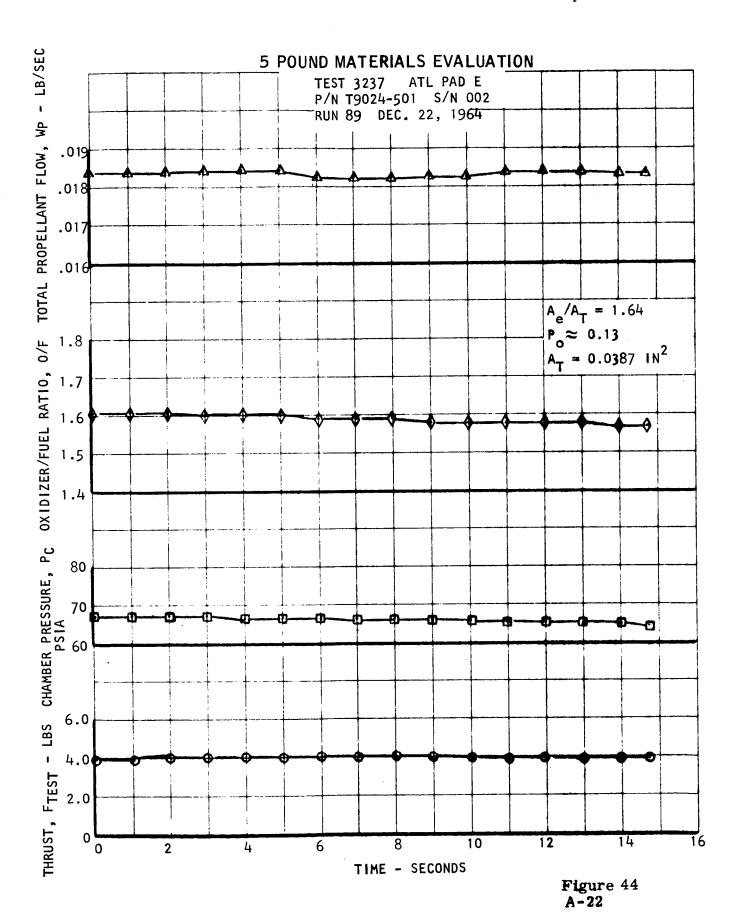
Jarquardi Jarquardi Van Nuys, California

DATE_____



COMBUSTION CHAMBER TEMPERATURE, TWALL - "F

Figure 43 A-21



DATE

1-25-65

THE Harquardt
COMPONITION VAN NUYS, CAL FORNIA

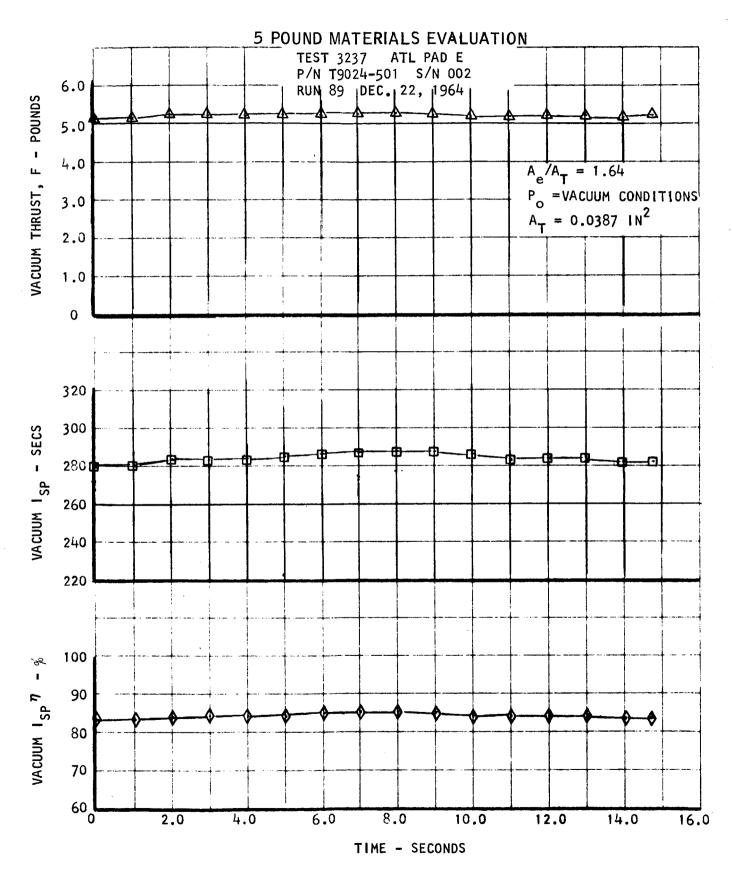
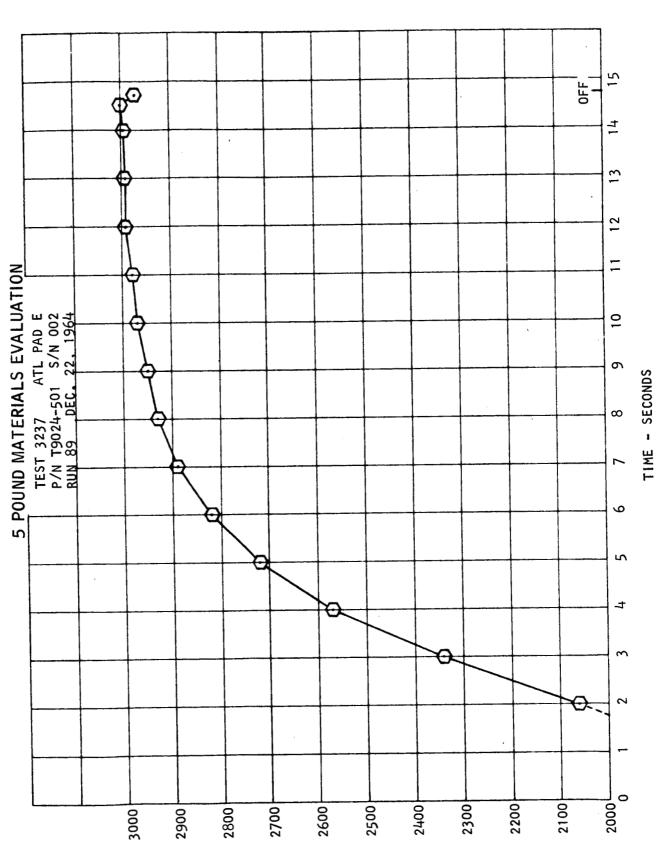


Figure 45 A-23

THE Sarguardt
CONTONITION VAN NUYS, CALIFORNIA

DATE.____



COMBUSTION CHAMBER TEMPERATURE, TWALL - "F

THE Harquardt

COMPONATION VAN NUYS, CALIFORNIA

DATE

TMC Report No. S-454

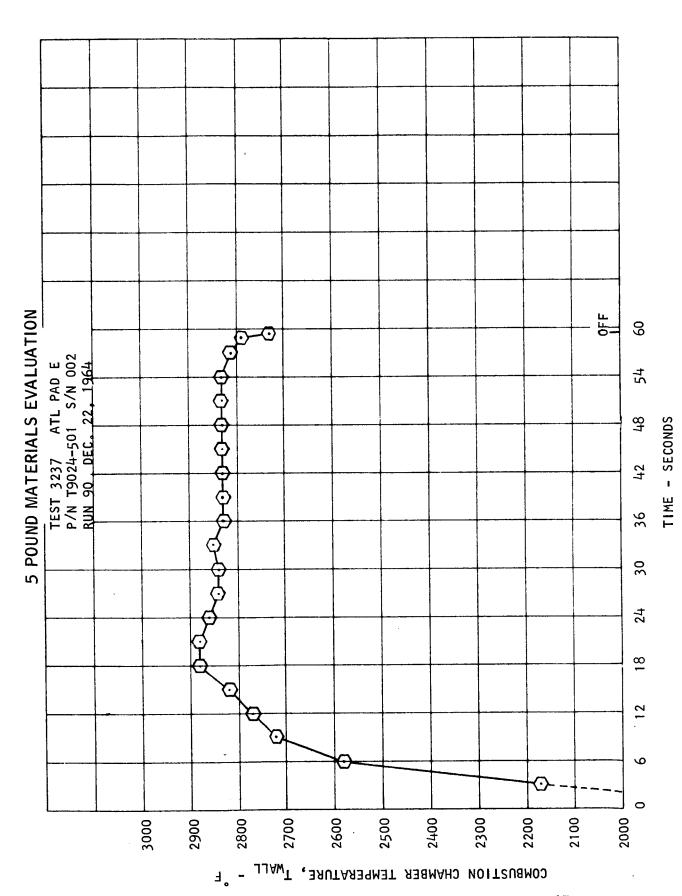
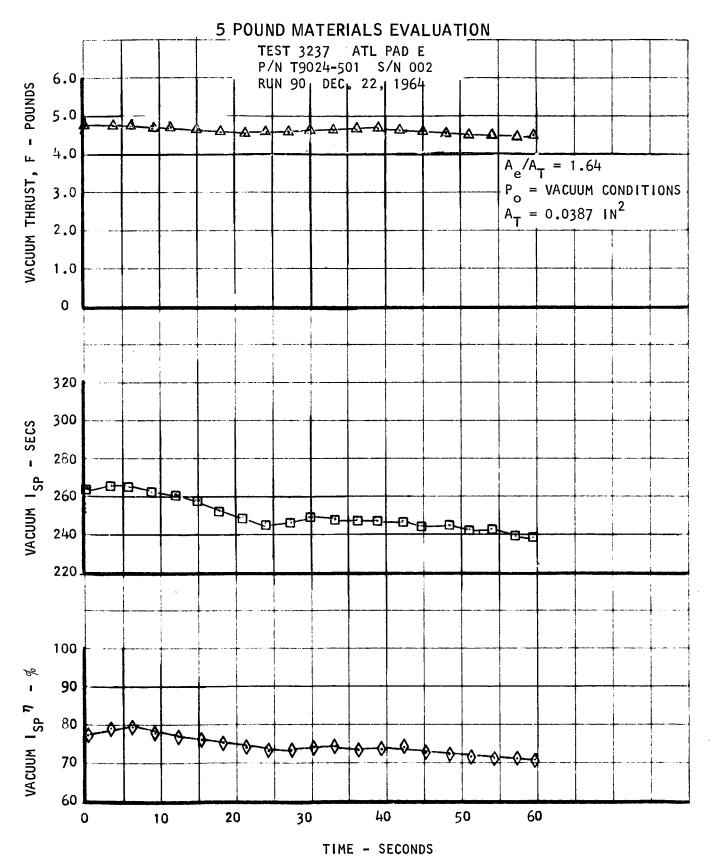


Figure 47 A-25 Jarquardt COMPONATION VAN NUYS. CAL FORNIA





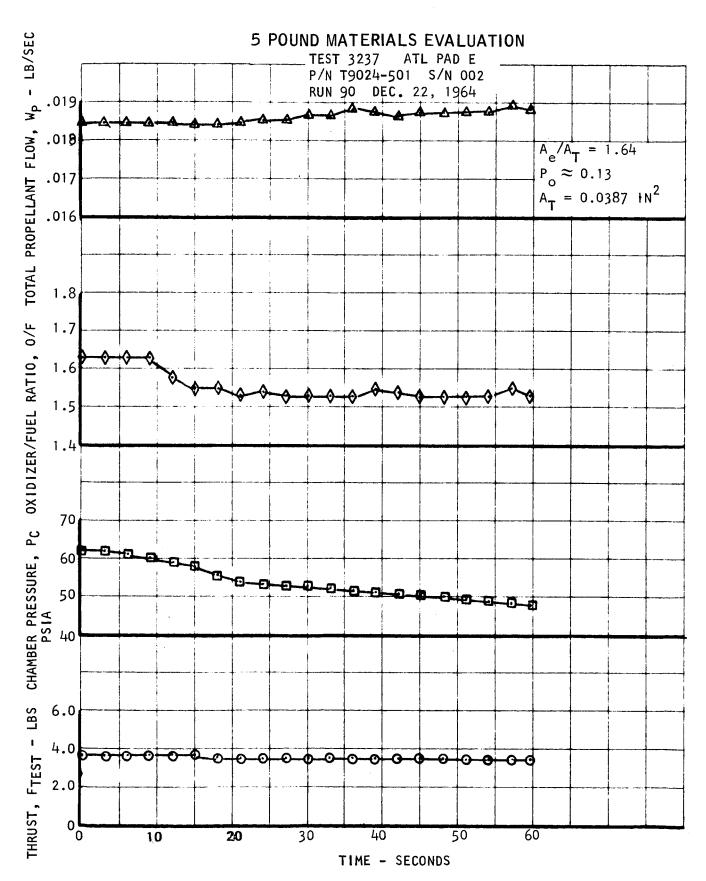


Figure 49 A-27

TMC Report No. S-454

DATE SUMMARY SHEETS

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135 .025 .025 .026 .026 80 .006 .011 1.63 2177 2320 864 & 566 335.9 2844 4.89 136 .035 .026 .026 810 .006 .071 1.63 220.6 2320 875 137 .023 .022 3.74 3.722 790 .006 810 .016 .071 1.63 220.6 2320 86	2300. 030 3.77 3.77.8. 790 . 050. 051.	.0107 .0172/.65 219.3 251.8 87.1	336.0 286.8		- 19
133 325 325 325 325 325 325 325 325 488	133 .035 .020 3.123.120 140 .0003	1.0106 .0/71 1.63 2/7.7 252.0 86.4	335.9 2844		62 —
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SUMMARY SHEET ERING TEST SECTION MATERIALS EVALUATION	521 S/N 002 EAKINE ASSY. TR	P. RAT C*, C*, C*, R I SP ISP	PSIA # FYEG PYEC 90 SEC. SEC.	72.5 2.81 5024 5703 88.1 337.0 294.9	72.5 2.81 5024 5703 88.1 3370 293.2	72.0 2.79 5005 5704 87.7 3370 294.9	720 2.79 5005 5706 87.9 336.0 294.0	72.0 2.79 5005 5706 87.9 736.6 213.3	72.0 2.79 5005 5704 87.9 336.0 294.0	72.0 2.79 5027 5706 88,1 3360 295.3	720 2.79 5021 5706 88,1 336,0 295.3	72.0 2.79 5027 5706 88.1 336.0 294.0		4 71. 68.8 3.88 1695 1716 59.5 83. 1.78	69.0 2.67 4787 5691 84.1 336.8 281.6	2.67 4787 5691 84.1 3368 280.2	69.0 2.67 4811 5696 84.5 336.8 284.2	69.0 2.67 4811 5696 84.5 336.8 283.6	69.0 2.67 4800 5697 84.3 33.8 282.9	69.0 2.67 4816 5697 845 3368 2846	ES32878 518 169 918h 19.2 069	
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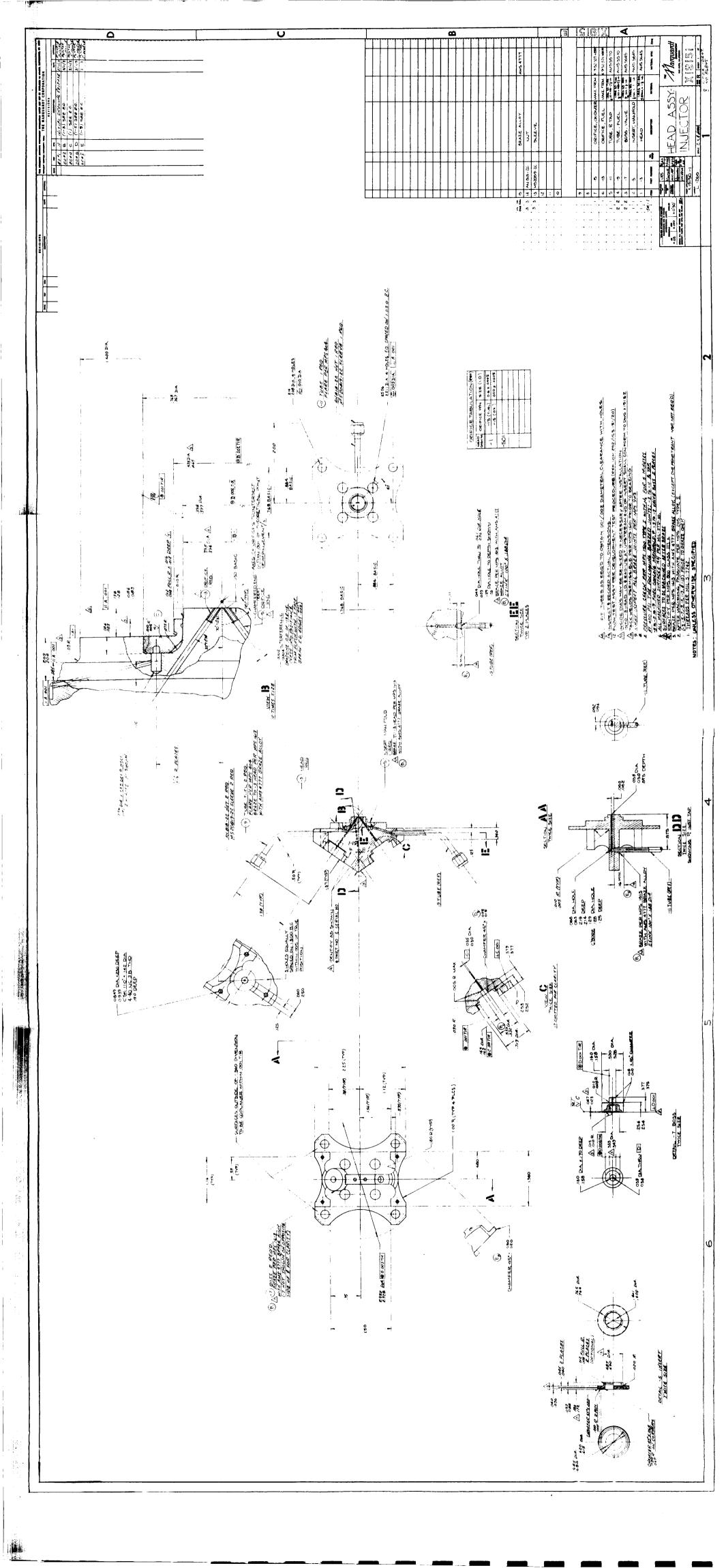
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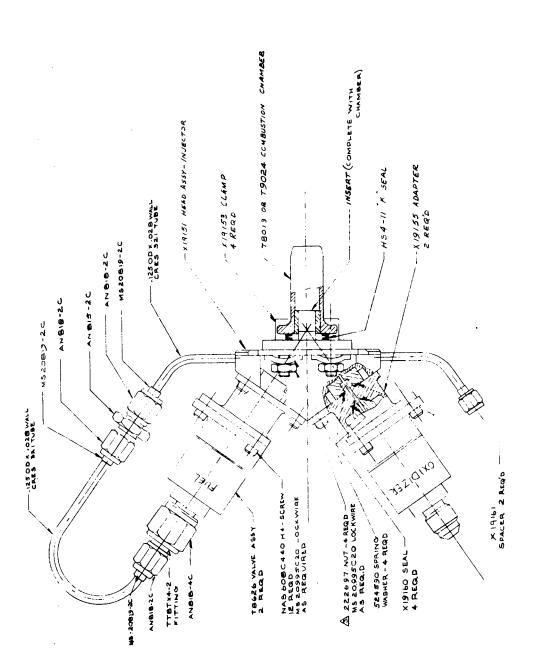
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TMC Report No. S-454

APPENDIX B

DESIGN DRAWINGS

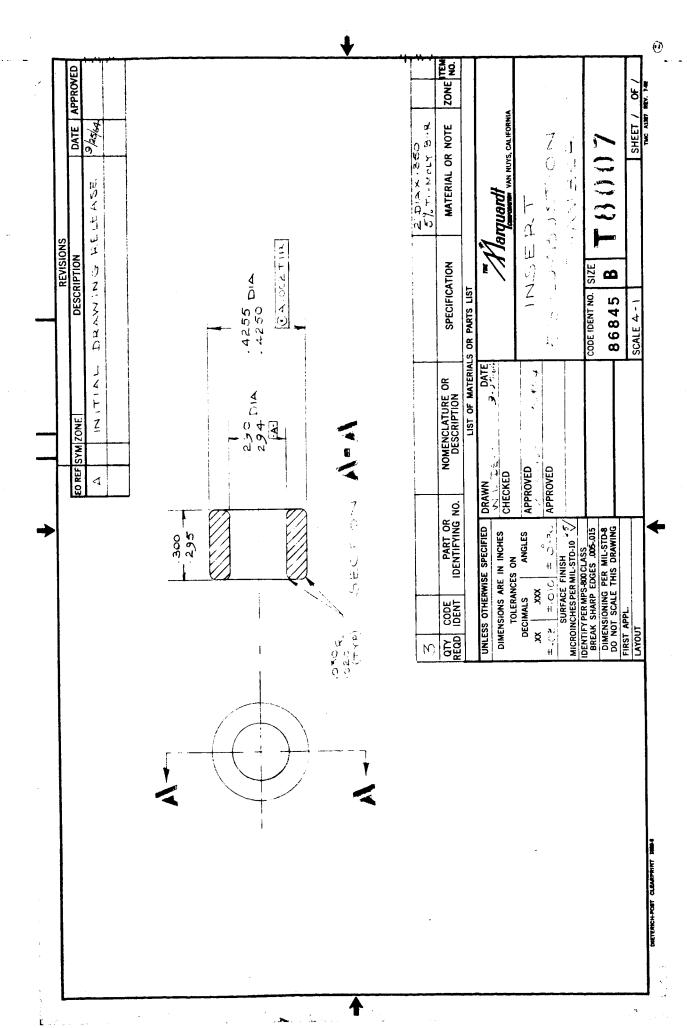


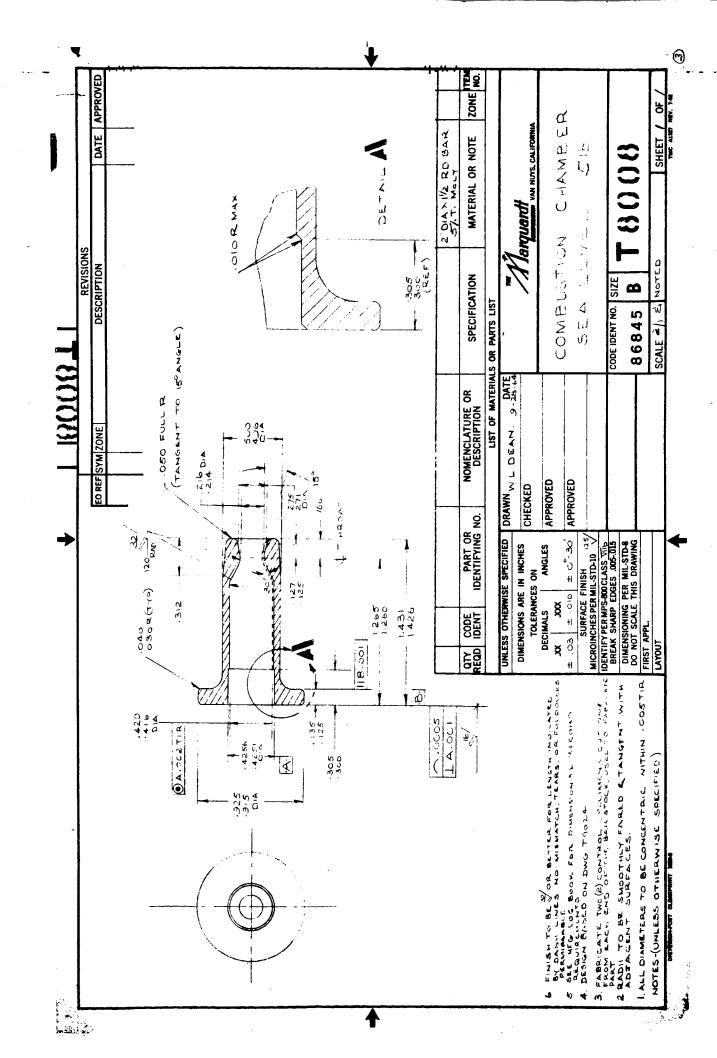


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4, PRESSURE CHECK ASSEMBLY USING DRY FILTERED	NITROGEN - NO LEAKAGE WHEN HELD AT A	PRESSURE OF 150 PSIG 1 SPSI DURING A PERIOD OF 2 MIN	
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Y. ASSEMBLY	LEAKAGE	150 PSIG #	
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PRESSURE	NITROGEN	PRESSURE	
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Mound	5 * ENGINE ASSY	5 8012	
	ν.	86845	7
CHECKED A 1/9/2	APROVED		





ENGINEERING EXPERIME	NTA. ORDER			NO. T 8013
FART NAME 516 COMBUSTION	ON CHAMBER	-/ئى-	Ti- Mory	ISSUE DATE
A.M.R. NO. (IF APPLICABLE)	DUE DATE 10-14-54-	ORIGINATED		10-13-54- MFG. CHARGE NO. BE-69-5-1
DELIVER TO:	PROJECT ENGINEER		SHOP SUPERVIS :R	SIGN WHEN COMPLETED)
ASSEME	BLE. THE	DA	सग ्ड	HOWN IN
T8007 INSERT	FIG	J/J/A	T	BOOB COMBINI CHAMBER
INTERFERENCE & INSURT O		いたごり	* JOMAHO	L RECESSED ID
INSERT OF	•	218 M 22 14 2	BER SE	MTEKPERUNCE
. 4255	250	425%	.4251	WAX . ODG
USING MAXIMUM	TEMP DIFF	= Coef	totex by	oria dia
	•	3. 1x10	204 -6 x .4250	= 320°F